ANALYTICAL ABSTRACTS

Dealing with all branches of Analytical Chemistry: Published Monthly with THE ANALYST by the Society for Analytical Chemistry

Editor: NORMAN EVERS, B.Sc., Ph.D., F.R.I.C.
20, EASTCHEAP, LONDON, E.C.3
Telephone: MANsion House 6608

Published for the Society by
W. HEFFER & SONS, LTD., CAMBRIDGE, ENGLAND

Volume I

Price 4s.

Subscription Rate, inclusive of Index, 50s. per annum, Post Free

No. 8, Abstracts 1748-2033

August, 1954

PRICE OF ANALYTICAL ABSTRACTS

Subscription Rate, inclusive of Index	50s. per annum, post free.
Abstracts printed on one side of the page only, exclusive of Index	60s. per annum, post free.

The subscription rate for *The Analyst* together with *Analytical Abstracts* and Indexes is 80s. per annum, post free.

All further enquiries about subscriptions should be made through the Secretary, the Society for Analytical Chemistry, 7-8, Idol Lane, London, E.C.3. Telephone: MANsion House 6608.

ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

1748. **Purification and properties of disodium salt of ethylenediaminetetra-acetic acid as a primary standard.** W. J. Blaedel and H. T. Knight (*Anal. Chem.*, 1954, **26** [4], 741–743).—A single recrystallisation from ethanol is sufficient for the purification of the reagent grade of dihydrated disodium salt of ethylenediaminetetra-acetic acid (**I**). The product is stable to heat and can be dried to the anhydrous form at temp. up to 150° C without appreciable decomposition. The dihydrate is the most suitable form for standards, the optimum temp. for safe air-drying being 80° C at 50 per cent. relative humidity. Standards of 0·01*M* **I** purified in the above manner change less than 0·05 per cent. in titre after 5 months' storage in borosilicate-glass or polyethylene bottles. G. P. Cook

1749. **Sodium metavanadate as volumetric reagent. I. Iodine monochloride method.** Balwant Singh and Ranjit Singh (*Anal. Chim. Acta*, 1954, **10** [5], 408–412).—A standard solution of sodium metavanadate affords quant. titrations in the presence of iodine monochloride according to the following reactions—

$$I' + 2VO_3' + 8H^{\cdot} \rightarrow I^{\cdot} + 2VO^{\cdot\cdot} + 4H_2O$$
$$AsO_3''' + 2VO_3' + 6H^{\cdot} \rightarrow AsO_4''' + 2VO^{\cdot\cdot} + 3H_2O$$
$$Hg^{\cdot} + VO_3' + 4H^{\cdot} \rightarrow Hg^{\cdot\cdot} + VO^{\cdot\cdot} + 2H_2O$$
$$CNS' + 6VO_3' + 16H^{\cdot} \rightarrow CN' + SO_4'' + 6VO^{\cdot\cdot} + 8H_2O$$
$$HSO_3' + 2VO_3' + 5H^{\cdot} \rightarrow SO_4'' + 2VO^{\cdot\cdot} + 3H_2O$$
$$SO_3'' + 2VO_3' + 6H^{\cdot} \rightarrow SO_4'' + 2VO^{\cdot\cdot} + 3H_2O$$
$$S_2O_3'' + 8VO_3' + 22H^{\cdot} \rightarrow 2SO_4'' + 8VO^{\cdot\cdot} + 11H_2O$$
$$Fe^{\cdot\cdot} + VO_3' + 4H^{\cdot} \rightarrow Fe^{\cdot\cdot\cdot} + VO^{\cdot\cdot} + 2H_2O$$
$$N_2H_4 + 4VO_3' + 12H^{\cdot} \rightarrow N_2 + 4VO^{\cdot\cdot} + 8H_2O$$

General procedure—To a suitable quantity of the substance in a stoppered flask, add 25 ml of water, 30 ml of conc. HCl, 5 ml of 0·02 *M* ICl and 5 ml of $CHCl_3$; titrate at room temp. with 0·1 *N* $NaVO_3$, shaking the mixture vigorously during the titration. The end-point is marked by a change from pale violet to pale yellow in the $CHCl_3$. More HCl is added, if necessary, during the titration to produce a final concn. of 7 to 7·5 *N*. W. C. Johnson

1750. **Complex metal tetraphenylboron salts.** W. Heyl (Brit. Pat. 705,719, 10.10.51. German Pat. 11.10.50).—Sodium tetraphenylboron, in which the benzene nucleus may also be substituted, is prepared by treating the corresponding phenyl magnesium halide with BF_3 and salting out the reaction mixture with NaCl. Sodium tetraphenylborons are excellent reagents for the quant. estimation of $K^{\cdot}$, $NH_4^{\cdot}$, $Rb^{\cdot}$ and $Cs^{\cdot}$, with which they form white finely crystalline precipitates of the corresponding complex salts even from very dil. solutions containing these ions. The ppt. are still more difficultly sol. than the corresponding perchlorates. J. M. Jacobs

1751. **Method for preparation of fluorine-low calcium oxide.** D. A. Weddle and R. L. Maurer (*Anal. Chem.*, 1954, **26** [3], 584–585).—One hundred g of CaO are slaked with 100 ml of water, the slurry is chilled and 300 ml of 72 per cent. $HClO_4$ are added. After filtration a further 300 ml of $HClO_4$ are added and any H_2SiF_6 formed is distilled in steam from an all-borosilicate glass apparatus until approx. 1·5 litres of distillate have been collected. The residue is cooled to $< 0°$ C and NH_3 is passed in. The pptd. NH_4ClO_4 is filtered and the Ca in the liquor is pptd. with saturated pure ammonium oxalate soln. The calcium oxalate is ignited to $CaCO_3$ or CaO (70 to 80 per cent. over-all recovery). The F′, which must be $< 0·2$ μg per g of CaO, is determined by the titrimetric $Th(NO_3)_4$ method. D. A. Pantony

1752. **Inorganic indicator applicable to acidimetric titrations.** A. Krause and M. J. Daleszyńska (*Roczn. Chem.*, 1954, **28** [1], 134–138).—When to an aq. soln. of $FeSO_4.7H_2O$ some H_2O_2 is added at 20° C, a yellow basic ferric sulphate is pptd. This is converted by OH′ into red *iso*-orthoferrihydroxide amorphous to X-rays. The coagulation of the ppt. is best achieved at the iso-electric point (pH = 8), which is identical with the acid - base neutralisation point. The basic ferric sulphate is a convenient indicator for titrations with NaOH of strong inorganic acids in dilutions to 0·01 *N*, and of strong organic acids like formic and acetic acids at dilutions to 0·1 *N*. The indicator, however, cannot be used to titrate bases with acids. To prepare the indicator, dissolve 3 g of $FeSO_4.7H_2O$ in 100 ml of distilled water at 20° C, filter, mix with 15 ml of H_2O_2; after 2 to 3 hr. filter the yellow coagulate, wash with distilled water to remove all $Fe^{\cdot\cdot\cdot}$ and SO_4'', and dry as much as possible on a Buchner funnel. The indicator is used in form of a pulp, which is obtained by thoroughly mixing the filter cake with twice its amount of water. Very accurate results are obtained in the titration of H_2SO_4, owing to the common anion; to titrate dil. HCl, some K_2SO_4 should be added to suppress the peptising influence of HCl. The indicator is not applicable to the titration of orthophosphoric, tartaric and citric acids, owing to the formation of complex compounds. Comparison with phenolphthalein and methyl orange indicators showed agreement to 1 per cent. for inorganic acids and approx. 4 per cent. for organic acids.
H. Burstin

1753. **Prediction of the curves of potentiometric determinations.** J. Coursier (*Anal. Chim. Acta*, 1954, **10** [2], 182–191).—Previous work has been described relating polarisation curves with redox potentials (*Brit. Abstr. C*, 1952, 577). It is now shown that a knowledge of the polarisation curves enables interpretation of experimental observations in potentiometric titrations. Examples presented are iodine - thiosulphate, As^{III} - iodine and Fe^{II} - $KMnO_4$ The use and choice of a "potential mediator" are also defined. H. F. W. Kirkpatrick

1754. **Prediction of bimetallic potentiometric determination curves by means of polarisation curves.** J. Coursier (*Anal. Chim. Acta*, 1954, **10** [3], 265–272).—The interpretation and prediction of potentiometric curves from a knowledge of the polarisation curves (*Anal. Abstr.*, 1954, **1**, 1753) is extended to the use of two different electrodes in various types of redox systems. A number of examples of the use of Pt - graphite electrodes is described together with the determination of Fe^{II} with V^{V} at Pt - Mo electrodes. H. F. W. KIRKPATRICK

1755. **Application of spectrophotometric end-point in volumetric analysis. Use of visible and ultra-violet region of the spectra.** P. B. Sweetser (*Dissert. Abstr.*, 1954, **14** [2], 232–233).—The following applications are considered: (*i*) estimation of mg amounts of As, Fe and U by spectrophotometric titration with 10^{-2} to 10^{-4} *M* $Ce(SO_4)_2$ at 320 mμ for As or 340 to 360 mμ for Fe and U soln. previously reduced with 90 per cent. Cd - Hg, (*ii*) bromate - bromide titrations in which the spectrophotometric end-point is given by the absorption of tribromide ion at 270 to 360 mμ, (*iii*) estimation of Fe, Cu and Ni in alloys and ores by spectrophotometric titration with 10^{-2} to 10^{-3} *M* Versenate soln. in the u.v., (*iv*) spectrophotometric end-points in Versenate titrations for estimation of Ca, Mg, Cd and Zn in NH_3 - NH_4Cl buffered soln., of Ca in presence of Mg and Cd in the presence of Zn in aq. NaOH, of Zr by titration of excess of Versenate with Fe^{III}, and of Ti by formation at low pH of a Ti - H_2O_2 - Versenate complex. W. J. BAKER

1756. **Flame photometry.** M. Pinta (*Chim. Anal.*, 1954, **36** [5], 126–130).—Flame-photometric procedures and their numerous analytical applications are reviewed generally; special reference is made to the rapid determination (within 1 min.) of alkali and alkaline-earth metals in concn. as low as 0·5 μg per ml in the same soln. Arrangements for effecting spectral excitation and for recording the spectra photographically or photo-electrically, with or without optical filters, are described. Factors affecting the analytical operations and their accuracy are discussed briefly; these include, variations in flame feed, strong background interference and perturbations caused by mol. interactions. W. J. BAKER

1757. **Some results of the analysis of elements by flame spectrophotometry.** M. Servigne with P. Guérin de Montgareuil (*Chim. Anal.*, 1954, **36** [5], 115–117).—The possibility of using the perturbations, caused by an element (Al), of the emission spectrum of another element (Ca) in order to determine the amount of interfering element in a soln. containing both is examined, and a flame-spectrophotometric procedure is developed for the rapid determination of 2 to 100 μg of Al per ml to within 3 per cent. Known amounts of Ca (as aq. $CaCl_2$) are added to aq. $Al(NO_3)_3$ and the percentage perturbation of the Ca line 4226A, caused by Al, is determined by comparison with aq. soln. of pure $CaCl_2$. The amount of Al is calculated from one of three standard curves for various concn. of Al. The limitations of the method are discussed, especially in relation to the permissible concn. of PO_4''', Cl', Ca'' and Sr'' in the Al soln. Fe and Cr do not interfere unless their concn. is > 100 μg per ml, when it is better to remove them initially from the soln. The procedure is regarded as a simple and rapid means of effecting series determinations of an element in a complex mixture, once the standard curve of element concn. has been drawn. W. J. BAKER

1758. **Coulometry: related phenomena of electrolysis and current-sweep polarography.** N. H. Furman (*J. Electrochem. Soc.*, 1954, **101** [1], 19c–22c).—Some recent developments that have made coulometry of increasing analytical usefulness are reviewed. 36 references. G. C. JONES

1759. **Minimum error polarographic analysis of binary mixtures.** A. Frisque, V. W. Meloche and I. Shain (*Anal. Chem.*, 1954, **26**, [3], 471–473).—Calculation of the concn. of components in a binary mixture of reversibly reducible compounds yielding no distinct separation of polarographic waves can be made by measuring the total diffusion current and the current at the potential of min. error. The single potential const., $K = 1\ [1 + 10^{(E-E_{\frac{1}{2}})\ n/0{\cdot}059}]$, can be calculated or determined by expt. at a chosen applied potential, and the diffusion current const. $K' = i_d/C$ measured for each component by expt. The concn. of *A* and *B* in mixture *AB* are then given by—

$$(A) = \frac{i_{dAB}\ K_B - i_{AB}}{(K_B - K_A)\ (K'_A)} \qquad (B) = \frac{(i_{AB} - i_{dAB}\ K_A)}{(K_B - K_A)\ (K'_B)}$$

The min. error potential is that at which $K_B - K_A$ is max., and, when *A* and *B* undergo the same electron change, it is given by $E = \frac{1}{2}\ (E_{\frac{1}{2}B} + E_{\frac{1}{2}A})$. The method was applied to mixtures of Tl^{I} and Pb^{II}, the results showing an average relative error of 3 per cent. H. F. W. KIRKPATRICK

See also Abstract 1884.

2.—INORGANIC ANALYSIS

1760. **Stoicheiometry of titration of metal ions with disodium salt of ethylenediaminetetra-acetic acid using high frequency technique.** W. J. Blaedel and H. T. Knight (*Anal. Chem.*, 1954, **26** [4], 743–746).—From data obtained by means of differential high-frequency procedures that ensure sharp end-points, it is shown that direct titrations of Cu^{II}, Zn, Ca and Mg with approx. 0·01 *M* disodium ethylenediaminetetra-acetate are stoicheiometric to within ≃ 0·1 per cent. under the conditions studied, *viz.*, various pH values, different concn. of NH_3, NaOH, etc. Titrations of 0·01 *M* $Cu(NO_3)_2$ and 0·01 *M* $ZnCl_2$ can be made with high accuracy in presence of conc. aq. NH_3 (pH 10) if a large excess of NH_3 is avoided. Titrations of 0·01 *M* $CaCl_2$ are accurate at pH > 8 in presence of aq. NH_3, NaOH or ethylenediamine, whilst 0·01 *M* $MgSO_4$ or $MgCl_2$ should be titrated at pH ≮ 10; $MgCl_2$ solutions give much sharper end-points than do those of $MgSO_4$. W. J. BAKER

1761. **The application of the complexones in metallurgical analysis. [II. Non-ferrous applications.]** E. G. Brown (*Metallurgia*, 1954, **49**, 151–155).—Specific proposals for the use of complexones in the analysis of non-ferrous metals, in particular Be, Bi, Ca, Fe, Mg, Mn, Ag, Tl and Zn, are surveyed. G. C. JONES

1762. **Determination of the humidity of a gas with Karl Fischer reagent.** C. Capitani and E. Milani (*Chim. e Ind.*, 1954, **36** [3], 177–183).—Full details are given (with dimensioned drawings) for the determination of the moisture content of gases, particularly of C_2H_2, by bubbling them through Karl

Fischer reagent contained in a suitably modified Meyer tube. The error is ± 2 per cent.
R. C. MURRAY

1763. **Analysis and determination of water by infra-red spectrometry.** J. Lecomte (*Chim. Anal.*, 1954, **36** [5], 118–122).—Infra-red spectrophotometric procedures for detecting and determining small amounts of H_2O in compounds, by using the characteristic bands between ≃ 2 and 6 μ, are reviewed, and use of the data to assess the physicochemical state of the water, as exemplified by the intermol. bonds of the mol., either with themselves or other mol., is explained. Determinations of < 10 p.p.m. of water vapour in Freon (at 2·67 μ, 10-cm cell) and ≮ 1 p.p.m. of liquid water in CCl_4 (at 1·89 μ, 20-cm cell), as well as studies of hydration and rehydration of opal, gelatin, montmorillonite, and so on, are possible. By using cells 0·3 to 1 mm thick and calibration curves based on the band at 2·65 to 3 μ, it is possible to determine 0·002 to 0·2 per cent. of H_2O in solvents (acetone, ether, pyridine, dioxan) with which the molecules of H_2O associate. Variations in intensity of the 1·6-μ band (owing to HOD) with H_2O concn. in D_2O enable the H_2O contents (if ≯ 4 per cent.) to be measured to within 0·1 per cent., although a preliminary calibration is necessary as Beer's law is not obeyed. The i.r. absorption methods of Thornton *et al.* (*Brit. Abstr. C*, 1950, 433) and Brown *et al.* (*Brit. Abstr. C*, 1951, 281) are also available for the analysis of D_2O. Displacements of the absorption max. are discussed generally in relation to zeolites, combined H_2O and H_2O of crystallisation, detection of OH′ and H_2O simultaneously, and the variable perturbatory effect of H_2O molecules on other ions or radicles present in hydrated compounds. This effect is studied best by extending the i.r. analysis to the bands between 6 and 15μ. In all instances the results should be co-ordinated with Raman spectra and thermogravimetric data. W. J. BAKER

1764. **Determination of lithium in spodumene by flame photometry.** R. J. Brumbaugh and W. E. Fanus (*Anal. Chem.*, 1954, **26** [3], 463–465).—A representative 0·5 g of ore (at 200 mesh) is ignited at 1900° F for 30 min., cooled and decomposed with 48 per cent. HF, fumed with H_2SO_4 and, together with the filtrate from a second similar treatment of the residue, is made up to 250 ml. The Li is determined hydrogen-flame photometrically by comparison with standards at 671 mμ. A precision of ± 0·74 per cent. is claimed. K^+, in 6-fold excess, Al^{+++} and H_2SO_4, within certain ranges of concn., and Fe^{+++} and Mg^{++} do not interfere, but Na^+ and Ca^{++} interfere seriously. D. A. PANTONY

1765. **Rapid determination of potassium in potassium salts by measurements of radiation.** K. C. Scheel (*Angew. Chem.*, 1954, **66** [4], 102–106).—A special form of Geiger - Müller counter is described for measurement of the β-radiation from ^{40}K in a method of determining K in minerals; it is suitable for use in the laboratory or outside, *e.g.*, in mines. Provided the material is of about 0·4-mm grain size, results of about the same order of accuracy as by the normal gravimetric method are obtained. R. C. MURRAY

1766. **Dissolving sodium - potassium alloys.** L. P. Pepkowitz (*Anal. Chem.*, 1954, **26** [3], 574).—A rapid and safe method for dissolving Na - K alloys is described; the alloys are cooled and cut on solid CO_2 and the pieces are dissolved in alcohol - solid CO_2 mixtures. D. A. PANTONY

1767. **Flame-photometric determination of sodium and potassium in zinc cadmium sulphide phosphors.** S. B. Deal (*Anal. Chem.*, 1954, **26** [3], 598–599).—Five g of zinc cadmium sulphide phosphor are dissolved in 10 ml of 70 per cent. $HClO_4$ with 10 to 15 ml of conc. HNO_3, and the soln. is evaporated to dryness and made up to 100 ml with H_2O. The Na (0·0001 to 0·001 per cent.) and K (0·001 to 0·02 per cent.) are determined flame photometrically by comparison with standards. D. A. PANTONY

1768. **The absorptiometric determination of traces of copper in highly purified water.** E. N. Jenkins (*Analyst*, 1954, **79**, 209–216).—A method is described for determining Cu in highly purified water. The Cu at concn. down to 0·001 p.p.m. is extracted as Cu^{II} diethyldithiocarbamate by a single extraction with $CHCl_3$ in presence of a citrate buffer and disodium ethylenediaminetetra-acetate and is measured absorptiometrically. Slight modification of the method is necessary in presence of Sb or Bi, but no interference results from presence of other common cations, S″ or CN′. The low concn. of Cu determinable by this method is significant in problems concerned with Al corrosion. A. O. JONES

1769. **Colorimetric determination of copper with neo-cuproine.** A. R. Gahler (*Anal. Chem.*, 1954, **26** [3], 577–579).—The sample is dissolved in a suitable solvent (aqua regia is preferred, but HF - HNO_3 - H_2SO_4 is required for ferrotungsten, HCl for scheelite and HCl - HF - H_2SO_4 - H_3PO_4 - $HClO_4$ for Co - Mo - W steels) and the soln. is evaporated to dryness or fumes. The residue is cooled and diluted to a standard vol. An aliquot containing 0·2 mg of Cu is treated with 5 ml of 10 per cent. $NH_2OH.HCl$ and 10 ml of 30 per cent. Na citrate. The pH is adjusted to 4 to 6 with aq. NH_3 and 10 ml of neocuproine reagent (0·1 per cent. of 2:9-dimethyl-1:10-phenanthroline in ethanol) and 10 ml of $CHCl_3$ are added and the Cu complex is extracted in a standard manner. The extract and washings are made up to 25 ml with ethanol and the absorption of the soln. is measured at 457 mμ with reference to a similarly treated blank soln. The Cu is determined by comparison with standards with an error of < 1 per cent. $HClO_4$, F′, NH_4^+, Nb^{+++++}, Ta^{+++++}, Mo^{+++++}, WO_4'' and VO_3' do not interfere, but traces of CN′ and S″ and high concn. of H_3PO_4 interfere seriously. D. A. PANTONY

1770. **Heterometric micro-titration of copper with oxine.** M. Bobtelsky and Y. Welwart (*Anal. Chim. Acta*, 1954, **10** [5], 459–463).—Heterometric titration (*Anal. Abstr.*, 1954, **1**, 901) is applied to the determination of Cu (0·3 to 1·6 mg).

Procedure—To the soln. of Cu^{++} (15 ml), add ethanol (2 to 4 ml) and *M* Na acetate (2 ml), and titrate from a micro-burette with 0·01 to 0·02 *M* 8-hydroxyquinoline in 50 per cent. ethanol. Plot an optical density curve for the course of the titration. Draw a line through the last portion of the curve; the intersection of this line with the abscissa representing the max. density indicates the end-point. Errors up to ± 1 per cent. are recorded.
W. C. JOHNSON

1771. **Direct heterometric micro-titration of copper with oxine in the presence of other metals.** M. Bobtelsky and Y. Welwart (*Anal. Chim. Acta*, 1954, **10** [5], 464–469).—The method in abstract 1770 above is extended to the determination of Cu^{++} in the presence of other metals. *Determination of Cu^{++} in the presence of Mg^{++}, Mn^{++}, Co^{++}, Ni^{++}, Al^{+++} or Cr^{+++}.*—To 18 ml of soln. containing ≃ 1 mg of Cu^{++}, add

2 ml of *M* Na citrate and titrate with 0·01 to 0·02 *M* 8-hydroxyquinoline in 50 per cent. ethanol. *Determination of Cu·· in the presence of Al···, Mn··, Cd·· or Pb··.*—To 16 ml of soln. containing ≃ 1 mg of Cu, add 2 ml of *M* K tartrate and 2 ml of *M* aq. NH_3, and titrate with 0·01 to 0·02 *M* 8-hydroxyquinoline in 50 per cent. ethanol. The accuracy of the titration is not affected by a 20-fold excess of $Mg^{··}$ or $Al^{···}$, a 50-fold excess of $Zn^{··}$, $Mn^{··}$, $Co^{··}$, $Ni^{··}$, $Cd^{··}$ or $Cr^{···}$, or a 150-fold excess of $Pb^{··}$, but with a large excess of $Zn^{··}$, $Al^{···}$, $Ni^{··}$ or $Cd^{··}$ it is necessary to have twice the previously specified concn. of $Cu^{··}$. W. C. JOHNSON

1772. **Copper alkyl xanthates in the gravimetric determination of copper and carbon disulphide.** G. Curli (*Chim. e Ind.*, 1954, **36** [3], 184–186).—The preparation, purification and thermal stability (loss in weight in 2 hr. at 100° to 105° C) of the Cu^{I} salts of methyl-, ethyl-, *n*-butyl-, *iso*pentyl-, decyl-, dodecyl-, hexadecyl- and benzylxanthic acids is reported. Only Cu decylxanthate shows no loss in weight and is therefore suitable for gravimetric determinations of Cu, although it is difficult to remove CS_2 from it. R. C. MURRAY

1773. **Spectrochemical determination of copper in turbine oils.** J. E. Barney, II (*Anal. Chem.*, 1954, **26** [3], 567–568).—Three to ten drops of turbine oil containing 1 to 50 p.p.m. of Cu are ashed on spectrograph electrodes, and the residue is mixed with graphite. The Cu is determined spectrographically by comparison with standards prepared from Cu naphthenate; the precision is ≃ 10 per cent. D. A. PANTONY

1774. **Photocolorimetric determination of silver with *p*-dimethylaminobenzylidenerhodanine.** M. Struszynski, T. Nowicka and Z. Marczenko (*Przem. Chem.*, 1953, **32** [11], 574–578).—The optical analytical properties of *p*-dimethylaminobenzylidenerhodanine have been investigated, including the influence of pH, time and temperature of reaction with Ag, and the effect of protective colloids on this reaction. A calibration curve is presented for 2 to 4-μg per ml amounts of the relevant silver compound. A Coleman spectrophotometer was used to determine the absorption, and the optical densities were measured on a photocolorimeter. The method is suitable for the determination of traces of Ag in ores. H. BURSTIN

1775. **Analysis of beryllium carbide.** S. A. Reed, E. S. Funston and W. L. Bridges (*Anal. Chim. Acta*, 1954, **10** [5], 429–442).—Beryllium carbide is prepared by heating BeO and lampblack; the analytical scheme provides for the determination of Be_2C, unreacted BeO and C, and traces of moisture and nitrides. *Free carbon* is determined by dissolving the sample in dil. H_2SO_4 (1 + 1), filtering, igniting at 900° C in a stream of O and absorbing the CO_2 in Ascarite. *Total beryllium* is determined in the filtrate from the free C determination. The pyrophosphate method of Cupr (*Brit. Abstr. A*, 1929, 285) is improved by a more precise control of pH. *Beryllium oxide* is determined by heating at 825° C in a stream of dried purified argon saturated with Br. A diagram of the apparatus is provided. The following reaction takes place: $Be_2C + BeO + 2Br_2 \rightarrow BeO + 2BeBr_2 + C$. The $BeBr_2$ is volatilised at the reaction temp., the C is eliminated by heating in a muffle furnace and the residual BeO is weighed. *Combined carbon* is evolved as CH_4 when Be_2C is dissolved in 85 per cent. H_3PO_4. The CH_4 mixed with O is passed over an electrically heated helix of nichrome or Co-plated Pt. Both these materials catalyse the combustion of CH_4 to CO_2. The former is operated at 850° to 900° C; the latter at 950° to 1000° C affords a more rapid conversion. A small furnace containing CuO oxidises any CO to CO_2. A diagram of the assembly is shown. The CO_2 is absorbed in a tared tube of Ascarite. *Nitrogen* is evolved as NH_3 by heating with 50 per cent. aq. NaOH. The NH_3 is steam-distilled into an excess of 0·01 *N* H_2SO_4 and determined by back-titration with 0·01 *N* NaOH. *Moisture* is determined by heating at 700° C in a stream of argon and collecting the evolved water in anhydrone. It is not possible to differentiate between combined (carbide) C and free (amorphous and graphitic) C by conventional combustion methods, but determinations of total C in an induction furnace verify the total calculated from the results given by the recommended methods. W. C. JOHNSON

1776. **Separation of beryllium from magnesium and magnesium from zinc as phosphates, and determination as pyrophosphate[s].** R. Airoldi (*Ann. Chim., Roma*, 1954, **44** [2], 127–133).—Be is separated by pptn. with $(NH_4)_2HPO_4$ at pH 5·3 to 5·5 from solutions containing up to 10 times the amount of Mg, and Mg from Zn with the same reagent in presence of NH_4Cl and much NH_3. The elements left in solution are later pptd. as double phosphate with an ammonium salt soln., and then calcined to pyrophosphates in the usual manner. The separations are nearly quantitative. R. C. MURRAY

1777. **Quantitative separation of the alkaline earths by ion-exchange chromatography.** M. Lerner and W. Rieman, III (*Anal. Chem.*, 1954, **26** [3], 610–611).—When a solution (⩾ 6 ml) of $Ba^{··}$, $Sr^{··}$ and $Ca^{··}$ at concn. up to 1 millimole of each as chlorides is passed through a column of colloidal Dowex 50 19 cm long and is eluted with 1·2 *M* ammonium lactate at a flow-rate of 0·56 cm per min., Ca appears quantitatively in the first 74 ml (excluding the first 6 ml), Sr in the next 58 ml and Ba in the third 325 ml of eluate. Ca can be then determined by oxalate pptn. and $KMnO_4$ titration, and Sr and Ba by gravimetric sulphate methods. D. A. PANTONY

1778. **Separation of magnesium from calcium by ion-exchange chromatography. Application to determination of calcium oxide and magnesia in limestones and dolomites.** D. N. Campbell and C. T. Kenner (*Anal. Chem.*, 1954, **26** [3], 560–561).—Ca (≃ 4·5 mg per 75 ml) and Mg (≃ 1·5 mg per 75 ml) are absorbed on a cation-exchange resin from a dil. HCl soln. and the Mg is eluted with 600 ml of 1·05 *M* HCl, after which it is determined by titration with disodium ethylenediaminetetra-acetate, with murexide as indicator. Separately, Ca plus Mg is determined in a similar titration, and the Ca concn. is found by difference. The mean deviation amounts to 0·01 mg of Mg; interferences in the titrations can be prevented by addition of KCN. D. A. PANTONY

1779. **Spectrochemical study of gravimetric procedure for barium, strontium and calcium.** P. R. Wilkinson, J. A. Gibson, jun., and A. J. W. Headlee (*Anal. Chem.*, 1954, **26** [4], 767–769).—The accuracy of the standard gravimetric methods of separating and determining Ba, Sr and Ca was examined spectrometrically by determining (*i*) the amounts of co-precipitants in $BaCrO_4$, Ca oxalate

and $SrSO_4$ ppt. and (*ii*) solubility losses of the respective elements in the filtrates. Different precipitating agents and various procedures were studied, the impurities in all ppt. and filtrate solids being determined spectrometrically against standards containing the impurities as carbonates. The material-balance values so obtained are used to correct the gravimetric results so that the latter give approx. theoretical values, particularly in the presence of low concn. of elements that generally co-ppt. wholly or in part with the major elements. The data are discussed in relation to the probable accuracy of the three gravimetric methods studied. W. J. BAKER

1780. **Amperometric titration of calcium with the disodium salt of ethylenediaminetetra-acetic acid.** H. A. Laitinen and R. F. Sympson (*Anal. Chem.*, 1954, **26** [3], 556–558).—In an H-cell fitted with calomel and dropping-mercury electrodes, a potential of $-1{\cdot}700$ V is maintained across a mixture of 50 ml of soln. containing > 2 mg of $Ca^{\cdot\cdot}$ and 10 ml of 0·01 *M* potassium zincate indicator. Standard Versene is run in under N, and the end-point is found by extrapolation of the current - titration curves. A precision of 0·6 per cent. is claimed. D. A. PANTONY

1781. **Determination of calcium in the presence of strontium by titration with disodium ethylenediaminetetra-acetate.** A. Rio and M. Todardo (*Ann. Chim., Roma*, 1954, **44** [2], 139–146).—Instead of the usual indirect determination of Ca with permanganate, the quicker procedure of finding the volume of standard NaOH required to neutralise the $H^{\cdot}$ liberated by addition of disodium ethylenediaminetetra-acetate (**I**) is proposed. In the presence of Sr, the total Ca and Sr is determined by titration with **I** and murexide at 8·6. Calcium is determined at pH 6·5 and the difference in titre gives the amount of Sr. R. C. MURRAY

1782. **Effects of anions on calcium flame emission in flame photometry.** G. L. Baker and L. H. Johnson (*Anal. Chem.*, 1954, **26** [3], 465–468).—At 554 mμ, ClO_4' increases Ca flame intensity, whereas AsO_4''', SO_4'', PO_4''' and Cr_2O_7'' decrease it. Only for mixtures of ClO_4' and Cr_2O_7'' is the effect additive; in mixtures of ClO_4' with SO_4'' and PO_4''' the depressant action of the latter anions predominates. P_2O_7'''' and S_2O_7'' are suggested as the active agents in decreasing the flame intensity. D. A. PANTONY

1783. **Colorimetric determination of strontium with chloranilic acid.** P. J. Lucchesi, S. Z. Lewin and J. E. Vance (*Anal. Chem.*, 1954, **26** [3], 521–523).—A $Sr^{\cdot\cdot}$ soln. is treated in an Amberlite IRA-410 ion-exchange column to remove $H^{\cdot}$, and to 5 ml of the eluate are added 5 ml of 0·05 per cent. chloranilic acid (2:5-dichloro-3:6-dihydroxy-*p*-quinone). After cooling in ice (3 to 12 hr.), the suspension is centrifuged and the absorption of the supernatant liquid is measured at 530 mμ with reference to a blank solution of reagent. The diminution of the absorption is a measure of the $Sr^{\cdot\cdot}$ concn.; the method is not recommended when cations other than $H^{\cdot}$ are present. D. A. PANTONY

1784. **The detection and determination of barium in table salt.** G. T. Muys (*Chem. Weekbl.*, 1954, **50** [6], 94–95).—Barium, derived from $BaCl_2$ used in removing sulphate, is present in some table salts; it may be detected and determined as follows. *Detection*—To 500 ml of filtered saturated soln. of the salt add, without stirring, 20 ml of H_2SO_4, sp. gr. 1·84, diluted (1 + 1) with water; after about 10 min. stir well; set aside overnight, decant the clear soln., and apply the flame test to the residue. *Determination*—In each of nine 500-ml Erlenmeyer flasks place 200 ml of filtered saturated soln. of Ba-free NaCl. To the flasks, add graduated quantities of standard $BaCl_2$ soln. (1 mg of $Ba^{\cdot\cdot}$ per ml), beginning with 0·6 ml and increasing in 0·2-ml steps to 2·2 ml; mix well. In two similar flasks place 200 ml of the test soln. prepared by filtering a saturated soln. of the sample at 20° C; to one, add 0·6 ml of standard $BaCl_2$ soln. and mix well. In a twelfth flask place 100 ml of test soln. and 100 ml of Ba-free NaCl soln. To each of the 12 flasks, add 4 ml of H_2SO_4 soln. [sp. gr. 1·84, dil. (1 + 1) with water], shaking after each addition. Fill 12 colorimeter tubes (30-mm diam., 135 mm to mark) with the solutions and place them on a black dish containing a shallow layer of water. After 45 min. compare the most suitable of the 3 test solutions (according to Ba content of sample) with the standards. Factors that affect the determination include the quantity of H_2SO_4 and the way it is added and variations in temp. The soln. must be mixed immediately the H_2SO_4 has been added. $Ca^{\cdot\cdot}$ does not interfere, provided the correct amount of H_2SO_4 is used. E. HAYES

1785. **Precipitation of barium chromate from homogeneous solution.** L. Gordon and F. H. Firsching (*Anal. Chem.*, 1954, **26** [4], 759–760).—Separation of $\simeq$ 0·1 g of Ba from Sr or Ca by pptn. as $BaCrO_4$ is effected in a hot soln. ($\simeq$ 170 ml containing 6 g of ammonium acetate, pH 1·7 to 1·8 before addition of $K_2Cr_2O_7$) in presence of urea (10 g), which slowly raises the pH to the required final value of $\simeq$ 5·7 so that co-pptn. of Sr is minimised and large easily filtered and easily washed crystals are formed. Only $\simeq$ 0·1 to 0·2 mg of Ba remains in soln. If $>$ 50 mg of Sr are present per 100 mg of Ba, a double pptn. is necessary, but a complete separation of Ba from Ca is possible with one pptn. and less rigorous control of pH. W. J. BAKER

1786. **The amperometric determination of zinc with potassium ferrocyanide. Use of rotating platinum anode.** T. D. Parks, O. D. Smith and S. B. Radding (*Anal. Chim. Acta*, 1954, **10** [5], 485–490).—Zinc (1·6 to 160 mg) is titrated with standard potassium ferrocyanide (0·0033 to 0·067 *M*) in the presence of NH_4Cl and HCl. A rotating platinum anode is used in conjunction with the Fisher Elecdropode, and a p.d. of 1 V is maintained between the anode and the S.C.E. The current, which is virtually constant until the $Zn^{\cdot\cdot}$ is completely pptd., shows a steep rise as soon as the ferrocyanide is in excess. W. C. JOHNSON

1787. **The simultaneous determination of cadmium and magnesium with disodium ethylenediaminetetra-acetate.** E. G. Brown and T. J. Hayes (*Analyst*, 1954, **79**, 220–224).—Simultaneous determination of Cd and Mg by titration with a solution of disodium ethylenediaminetetra-acetate containing $ZnSO_4$ is described. By selective control of the pH at 6·8 by means of a Na maleate - maleic acid buffer, the Cd alone is titrated. Mg is subsequently titrated at pH 10 after addition of an NH_4Cl - aq. NH_3 buffer solution. Solochrome black is used as indicator in both titrations. The mol. ratio of Mg to Cd must not exceed 1. Methods of overcoming interference by Fe are suggested; Ca and possibly Pb are titrated with the Cd. Interference by Zn cannot apparently be obviated. A. O. JONES

1788. **Ultra-violet spectrophotometric determination of mercury as mercuric thiocyanate complex.** G. E. Markle and D. F. Boltz (*Anal. Chem.*, 1954, **26** [3], 447–449).—The $Hg^{\cdot\cdot}$ soln. (0·1 to 3·0 mg per 100 ml) is treated with excess of 5 per cent. KCNS and the absorption of the $Hg(CNS)_4''$ is measured either at 281 mμ or, after extraction with *n*-butanol, at 286 mμ. A precision of 1·5 to 2·0 per cent. is claimed. NO_3', NO_2', Cr_2O_7'', VO_3', $Cu^{\cdot\cdot}$, $Co^{\cdot\cdot}$, $Fe^{\cdot\cdot}$, $Fe^{\cdot\cdot\cdot}$, $Pb^{\cdot\cdot}$, $Sn^{\cdot\cdot\cdot\cdot}$ and $Bi^{\cdot\cdot\cdot}$ interfere, but $NH_4^{\cdot}$, $Cd^{\cdot\cdot}$, $Mg^{\cdot\cdot}$, $Mn^{\cdot\cdot}$, $K^{\cdot}$, $Na^{\cdot}$, $Zn^{\cdot\cdot}$, Cl', MoO_4'', ClO_4', PO_4''', SO_4'' and WO_4'' do not, unless present in high concentration. D. A. PANTONY

1789. **Determination of mercury in the atmosphere. Submicro-analytical determination of mercuric ion in bromine and chlorine water based on its catalytic action.** S. Ašperger and I. Murati (*Anal. Chem.*, 1954, **26** [3], 543–545).—Atmospheric Hg is collected by absorption in Br water or in a liquid-air trap, the Br is removed by evaporation and the residue is diluted to give a $Hg^{\cdot\cdot}$ concn. of 10^{-5} to 10^{-7} mole per litre. Aq. nitrosobenzene (0·0666 per cent) is added, the pH is adjusted to 3·5, and, at 20° ± 0·05° C, a small volume of $K_4Fe(CN)_6$ reagent is added. The extinction is measured at 405 mμ after 30 min. and compared with that of a standard prepared in a similar manner. D. A. PANTONY

1790. **Polarographic study of [mercuric] mercury in the presence of ethylenediaminetetra-acetic acid. II. Amperometric titration of cations by complexone, based upon the anodic depolarisation wave.** G. Michel (*Anal. Chim. Acta*, 1954, **10** [1], 87–96).—The work described in part I (*Anal. Chim. Acta*, 1953, **9**, 184) is extended to amperometric titration of various cations. Solutions buffered at pH 6·4 containing a const. concn. of complexone (10^{-4} to 10^{-3} *M*) and known concn. of cation were polarographed, and the amperometric curve was obtained from the values of the limiting currents. Curves were normal with $Mn^{\cdot\cdot}$, $Co^{\cdot\cdot}$, $Ni^{\cdot\cdot}$, $Hg^{\cdot\cdot}$ and $Zn^{\cdot\cdot}$, but the alkaline-earth cations gave higher waves than expected during titration. It is assumed that dissociation in the region of the electrode of the less-stable complexes formed by the alkaline-earth cations contributes to the measured current. H. F. W. KIRKPATRICK

1791. **Determination of boron in boron - carbon film resistors.** I. G. Young (*Anal. Chem.*, 1954, **26** [4], 753–754).—The determination of B in the coatings of boron - carbon ceramic resistors is effected by heating a number of clean dry resistors (equiv. to 200 to 500 μg of B) with conc. H_2SO_4 (3 ml) and conc. HNO_3 (5 to 10 drops) for ≃ 5 min., washing, drying and weighing the resistors, and titrating the soln. (diluted to ≃ 60 ml and adjusted to pH 7 to 7·3) with 0·015 *N* NaOH in presence of mannitol and pH electrodes. Carbonates should be absent, and a correction for interfering cations is made by means of a blank titration on similarly treated uncoated ceramics. Precision and accuracy of the method are within ± 5 per cent. W. J. BAKER

1792. **The photometric determination of traces of aluminium in zinc.** E. F. Pellowe and F. R. F. Hardy (*Analyst*, 1954, **79**, 225–229).—A rapid method is described for the determination of 0·001 to 0·01 per cent. of Al in Zn by means of aluminon (ammonium aurinetricarboxylate). The metal is dissolved in HCl, most of which is then removed, and thioglycollic acid is added as complexing agent to prevent interference by Fe, Cu and Sn. The colour developed at pH 5·07 is measured absorptiometrically at ≃ 545 mμ, and the Al content is ascertained from a calibration graph. The method can be modified for determination of Al in certain Sb and Cd alloys. A. O. JONES

1793. **Spectrophotometric determination of aluminium in steel.** H. Arnfelt, S. Freyschuss and B. Rönnholm (*Jernkontorets Ann.*, 1953, **137**, 819–826).—Tests were carried out with Wiberley and Basset's method (*Brit. Abstr. C*, 1949, 373) of determining Al with 8-hydroxyquinoline. It was found possible to correct for the interference of small amounts of Fe by measuring the optical density of $CHCl_3$ extracts of Al and Fe - 8-hydroxyquinoline complexes at 390 and 470 mμ. CHEM. ABSTR.

1794. **Spectrophotometric determination of aluminium with particular regard to its determination in iron and steel.** B. Rönnholm (*Jernkontorets Ann.*, 1953, **137**, 827–848).—Spectrophotometric methods of determining Al are reviewed and an extensive bibliography is appended. N. E.

1795. **Volumetric determination of gallium.** O. W. Rollins and C. K. Deischer (*Anal. Chem.*, 1954, **26** [4], 769–770).—The method involves quant. pptn. of Ga^{III} with 8-hydroxyquinoline at pH 3·6 and 18° C, warming (steam-bath) for 1 hr., and solution of the washed ppt. in hot 1·5 *N* HCl, followed by titration of the dil. soln. with aq. bromate - bromide (≃ 0·1 *N* $KBrO_3$) until the indicator (methyl red) is decolorised. A slight excess of bromate - bromide soln. is then added, followed immediately by 4 g of solid KI in ≃ 20 ml of H_2O (the flask is kept stoppered), and the liberated I is titrated with $Na_2S_2O_3$ soln. by use of starch as indicator. The sample (100 ml) should contain 0·01 to 0·03 g of Ga^{III} freed from metals (Zn, Fe, Co, Ni, Cu and Al) that are quant. pptd. with 8-hydroxyquinoline. The error is ± 0·02 per cent. W. J. BAKER

1796. **Separation and microscopical identification of gallium in the presence of aluminium.** A. Wold, J. H. Baird and C. R. Hough (*Anal. Chem.*, 1954, **26** [3], 546–548).—$Ga(OH)_3$ and $Al(OH)_3$ dissolve in ammoniacal acetylacetone, which, on evaporation, leaves the acetylacetonates in similar monoclinic forms; fusion or recrystallisation from ethyl acetate converts the Ga compound to an orthorhombic form. Only Ga acetylacetonate (90 per cent. yield) is extracted by ether from 6 *N* HCl soln. D. A. PANTONY

1797. **The separation factor. A criterion for evaluation of fractional separation processes [of rare earths].** B. Weaver (*Anal. Chem.*, 1954, **26** [3], 474–475).—The separation factor is defined as the quotient of the ratios of 2 elements in two fractions derived by separation of a mixture of those elements. This concept is applied to determination of the efficiency of published methods for separating Hf - Zr and rare-earth mixtures. D. A. PANTONY

1798. **Fractional separation of rare earths by precipitation with mandelic acid.** B. Weaver (*Anal. Chem.*, 1954, **26** [3], 476–478).—La, Nd and Sm oxides and Sm and Gd concentrates are dissolved in HCl; at carefully controlled pH, and preferably in the cold, 16 per cent. mandelic acid is added. Occasionally, solution in HCl and pptn. in aq. NH_3 is repeated as many as 30 times, without further addition of the reagent. Considerable enrichment of individual rare earths is stated to occur. In particular, separation of Nd into the most soluble fractions is almost complete. D. A. PANTONY

1799. **Fractional separation of rare earths by oxalate precipitation from homogeneous solution.** B. Weaver (*Anal. Chem.*, 1954, **26** [3], 479–480).—One g of mixed rare-earth oxides is converted to chlorides and dissolved in 100 ml of H_2O; 0·5 g (approx. 0·5 equivalents) of methyl oxalate in 100 ml of H_2O is added. Hydrolysis of the ester and pptn. of the oxalates is allowed to proceed at 75° to 90° C (1 hr.) or, preferably, at 20° to 35° C (overnight). Comparison of separation factors with those of other procedures shows that the method offers no great advantages, except perhaps for the separation of Y.

D. A. PANTONY

1800. **Determination of small amounts of niobium in pure tantalum and its oxide.** J. Hastings and T. A. McClarity (*Anal. Chem.*, 1954, **26** [4], 683–685).—In the spectrophotometric determination of 0·01 to 0·3 per cent. of Nb in Ta metal, the sample (1 g) is dissolved in 48 per cent. HF (10 ml) in presence of 5 ml of aq. 0·5 per cent. $KHSO_4$ plus 0·15 per cent. oxalic acid (to compensate for soln. used in obtaining the calibration curve). After solution of the metal, the soln. is evaporated to incipient crystallisation, dil. (1 + 1) HCl (25 ml) is added, followed in quick succession by aq. 4 per cent. H_3BO_3 (25 ml), 20 per cent. $SnCl_2$ in HCl (10 ml), aq. 23 per cent. NH_4CNS (25 ml) and ether (25 ml). After shaking the mixture, separation of the ether layer and addition to it of a number of ether extracts of the aq. layer, the intensity of the yellow niobium - thiocyanate complex is measured spectrophotometrically at 386 mμ in 1-cm cells with ether as reference. Measurement is also made at 468 mμ if Mo is present, a correction then being applied for this. Concn. of reagents, manipulation and timing should be carefully controlled.

W. J. BAKER

1801. **Determination of carbon in tantalum.** A. F. Torrisi, J. L. Kernahan and R. E. Fryxell (*Anal. Chem.*, 1954, **26** [4], 733–734).—From 0·002 to 0·007 per cent. of C in Ta metal is determined by means of the low-pressure combustion apparatus used for steels (*Brit. Abstr. C*, 1945, 62), with addition of an interchangeable sample-holder for non-magnetic materials. The samples (0·5 g) are normally dropped successively into the platinum crucible (without Al_2O_3 liner) at 1100° C, but, if more than four samples are used, a steel sample should be burned after every two or three Ta samples so as to form a low surface-area Fe - Ta slag in the crucible; this eliminates de-gassing troubles. The results are claimed to be accurate to within 0·001 per cent.

W. J. BAKER

1802. **Continuous conductimetric determination of small amounts of carbon dioxide in gases.** J. Minczewski, J. Waclawik and A. Woroniecki (*Przem. Chem.*, 1953, **32** [12], 651–655).—The electric conductivity of a dil. $Ba(OH)_2$ soln. and its variations caused by reaction with CO_2 is used as a principle for continuous measurement of CO_2 (0·005 to 0·04 vol. per cent.) in industrial gases. A suitable laboratory apparatus is described and schematic sketches and test charts are reproduced. The apparatus has 2 separate sections, one being the calibration and the other the measuring device. They are connected with each other by a system of glass tubes and cocks. The main features of the measuring device are 2 glass cylinders with nickel electrodes, which are put into the circuit of a Wheatstone bridge. The $Ba(OH)_2$ soln. is passed first through one conductimetric cylinder, then brought into reaction with the gas stream containing CO_2, and finally passed through the other cylinder. The difference between the electrical resistances of the $Ba(OH)_2$ solutions in the two cylinders indicates the CO_2 content of the gas. The current is provided by an oscillator of 1000 c.p.s. A valve amplifier, which also serves as a rectifier, is put into the circuit in place of the telephone receiver of the bridge. The variations of the current are recorded automatically. A continuous and steady flow of both gas and $Ba(OH)_2$ soln. is essential for correct results.

H. BURSTIN

1803. **Spectrochemical determination of germanium in silicates.** P. G. Harris (*Anal. Chem.*, 1954, **26** [4], 737–739).—Concn. of $\simeq$ 1 p.p.m. of Ge in mineral samples of $<$ 0·5 g are determined by controlled distillation of $GeCl_4$ (after an initial HF - H_2SO_4 treatment), followed by co-pptn. of Ge and Sn sulphides ($\simeq$ 500 μg of Sn are used as carrier), solution of the centrifuged ppt. in aq. NH_3 and 6 per cent. H_2O_2 (3 drops of each), and excitation of the spectra of the resultant soln. in the cathode arc (7 amp., 200 V, 30 sec.). Separations for the lines Ge 3039·6 and Sn 3032·77 A are referred to concn. curves (prepared from standard soln.) for 0·1 to 2 μg of Ge; the sensitivity is $\simeq$ 0·05 μg with reproducibility of $\pm$ 10 per cent. Results for pitchstones and flue dusts are reported.

W. J. BAKER

1804. **Benzilic acid as a reagent in gravimetric determination of zirconium.** J. J. Klingenberg, P. N. Vlannes and M. G. Mendel (*Anal. Chem.*, 1954, **26** [4], 754–756).—An improvement on the procedure developed previously by Venkataramaniah *et al.* (*Brit. Abstr. C*, 1952, 5) for gravimetric determination of Zr is described. To the soln. ($\simeq$ 35 ml) containing 0·001 to 0·06 g of Zr are added 15 *N* HCl (15 ml), the mixture is heated to 100° C, hot aq. 2 per cent. benzilic acid (100 ml) is added, the voluminous ppt. is digested at 95° C for $\simeq$ 10 min. (to effect granulation) and is then filtered, washed with hot H_2O, dried and ignited at 1000° C in a porcelain or platinum crucible. Direct weighing of the Zr benzilate ppt. is not possible. Benzilic acid in presence of 10 per cent. HCl inhibits pptn. of other ions normally associated with Zr.

W. J. BAKER

1805. **Determination of small amounts of tin with dithiol.** M. Farnsworth and J. Pekola (*Anal. Chem.*, 1954, **26** [4], 735–737).—The procedure permits the accurate determination of $>$ 16 μg Sn per ml in biological or other samples. After destruction of any org. matter, As, Sb, etc., are removed by distillation as chlorides and Sn is then separated completely by distillation with a mixture of aq. HCl and HBr (1 + 3) at 137° to 147° C. The Sn soln. (free from interfering elements) is treated with H_2SO_4 and H_2O_2 to remove Br, Sn^{IV} is reduced with thioglycollic acid, and the coloured Sn - dithiol complex is developed in presence of 30 per cent. Santomerse S as dispersing agent. The light transmittance of a portion of the final soln. (50 ml containing 1·5 $\pm$ 0·25 ml of conc. H_2SO_4) is measured at 530 mμ on a Beckman spectrophotometer. The calibration curves are prepared from standard Sn soln.

W. J. BAKER

1806. **New colorimetric method for estimation of tin in copper alloys and plated articles.** J. Davis and A. J. Easton (*Lab. Practice*, 1954, **3** [5], 190).—Determination of 0·1 to 70 per cent. of Sn in Cu alloys, to within 2 per cent., is effected in soln. containing 0·1 to 0·7 mg of Sn by the removal with

$CHCl_3$ of the brown Sn complex with Na diethyldithiocarbamate (1 per cent. soln. in aq. NH_3) after the pH of the soln. has been adjusted to 9·6 so that the Sn complex remains in the aq. phase. Dil. HCl is then added to the aq. phase until the pH is between 5·8 and 6·0, and the yellow Sn complex is extracted with $CHCl_3$ (into which it passes at the lower pH). The extract is dried over anhydrous $CaCl_2$, diluted to 10 ml with $CHCl_3$, and the intensity of colour is measured (440-mμ filter) against calibration curves prepared by use of pure Sn. For alloys containing $<$ 5 per cent. of Sn, most of the Cu should be separated electrolytically before applying the above procedure. W. J. BAKER

1807. **A new method for the potentiometric determination of lead with alkaline permanganate.** I. M. Issa, R. M. Issa and A. A. Abdul Azim (*Anal. Chim. Acta*, 1954, **10** [5], 474–484).—The reaction between $Pb^{\cdot\cdot}$ and $KMnO_4$ in alkaline soln. is investigated under various conditions. For its determination, $Pb^{\cdot\cdot}$ is taken into soln. as sodium plumbite, the soln. is made *N* in respect of NaOH and added slowly with stirring to an excess of standard $KMnO_4$ in *N* NaOH containing $Ba(NO_3)_2$. The excess of $KMnO_4$ is titrated potentiometrically with a standard $Pb^{\cdot\cdot}$ or sodium plumbite soln. in *N* NaOH in the presence of ZnO and HgO as catalysts.
W. C. JOHNSON

1808. **Automatic spectrophotometric titrations. Determination of milligram quantities of thorium.** H. V. Malmstadt and E. C. Gohrbrandt (*Anal. Chem.*, 1954, **26** [3], 442–445).—$Cu^{\cdot\cdot}$ and $Th^{\cdot\cdot\cdot\cdot}$ are titrated directly with standard disodium ethylenediaminetetra-acetate, the end-points being detected spectrophotometrically at 320 mμ (pH 4·2) and 290 mμ (pH 3·1), respectively. Alternatively, $Th^{\cdot\cdot\cdot\cdot}$ may be determined by addition of excess of Versenate, followed by a back-titration with standard $Cu^{\cdot\cdot}$. Change of absorption with addition of Versenate is recorded automatically in a specially designed cell with a modified spectrophotometer. $Pb^{\cdot\cdot}$, $Cu^{\cdot\cdot}$, $Ni^{\cdot\cdot}$, $Bi^{\cdot\cdot\cdot}$, $Be^{\cdot\cdot}$, $ZrO^{\cdot\cdot}$, $Sn^{\cdot\cdot}$, $Sn^{\cdot\cdot\cdot\cdot}$ and $Ce^{\cdot\cdot\cdot}$ interfere. A precision of better than 0·5 per cent. is claimed for determination of 20 to 70 mg of $Th^{\cdot\cdot\cdot\cdot}$. D. A. PANTONY

1809. **Determination of nitrogen content of noble gases. Absorption in titanium metal.** H. S. Dombrowski (*Anal. Chem.*, 1954, **26** [3], 526–528).—Six hundred litres of A or He containing 1 to 10 p.p.m. of N are passed through 10 g of Ti sponge (8 to 20 mesh and of known N content) at 1050° C. After cooling, the combined N is determined by a modified Kjeldahl method. D. A. PANTONY

1810. **Kjeldahl method as applied to determination of nitrogen in nitrates.** W. E. Dickinson (*Anal. Chem.*, 1954, **26** [4], 777–779).—The accuracy of the modified Kjeldahl methods for determining N in materials of high nitrate content is examined experimentally. The results show that the Gunning variation of the standard method adopted by the Association of Official Agricultural Chemists ("Methods of Analysis," 1950) yields N values that are $\simeq$ 0·2 per cent. too low unless a 0·35-g sample is taken or the salicylic acid is increased from 1 to 2 g per 30 ml of H_2SO_4. If this is done, the A.O.A.C. standard method and the modified Gunning method should give accurate results for fertilisers and most inorganic N compounds. For the latter, or for nitrates only, a recommended modified Kjeldahl procedure involves (*i*) solution of the sample (0·7 g) in H_2SO_4 (30 ml) and salicylic acid (2 g) to form the very stable nitrosalicylic acid, (*ii*) reduction of the nitro compound with anhydrous $Na_2S_2O_3$ (3 to 4 g) in hot soln., followed by (*iii*) digestion (1 to 2 hr. at $\simeq$ 90° C) of the amino compound with anhydrous Na_2SO_4 (15 g) to form $(NH_4)_2SO_4$. To the diluted soln. is added powdered Zn (0·05 g) and an excess of aq. NaOH, and the soln. is distilled into an excess of 0·5 *N* H_2SO_4 and back-titrated with 0·1 *N* NaOH. Interfering substances are the same as in the classical Kjeldahl method. Results for N in $NaNO_3$ are precise and accurate, ranging from 16·40 to 16·48 per cent. W. J. BAKER

1811. **Identification of hydroxylamine and hydrazine by paper chromatography.** J. M. Bremner (*Analyst*, 1954, **79**, 198–201).—A method for separation and identification of μg amounts of NH_2OH and N_2H_4 is described. It involves ascending paper chromatography with acidic solvents and identification by R_F values and by spraying the dry chromatogram with detecting reagents, such as picryl chloride, ammoniacal diacetylmonoxime Ni salt, ninhydrin and modifications of Csaky's reagents (*Brit. Abstr. C*, 1949, 345). The solvents used with most success were methanol, ethanol or butanol containing various amounts of HCl.
A. O. JONES

1812. **Photometric determination of phosphorus in steel.** N. Bäckström (*Jernkontorets Ann.*, 1953, **137**, 849–853).—The sample is dissolved in 7·5 *N* HNO_3 containing $HClO_4$, and evaporated to $HClO_4$ fumes. It is reduced in hot soln. with Na_2SO_3. The colour of molybdenum blue is produced in cold soln. by adding $(NH_4)_2MoO_4$ and $SnCl_2$ with sulphamic acid as a stabiliser. Proper acidity must be maintained during both the reduction and the development of the colour. The light absorption of the molybdenum-blue complex is measured on a photo-electric absorptiometer. CHEM. ABSTR.

1813. **Determination of micro-amounts of phosphorus in high-purity iron.** O. R. Gates (*Anal. Chem.*, 1954 **26** [4], 730–732).—Determination of $\simeq$ 0·0001 per cent. of P in a 4-g sample of high-purity iron is effected by separating most of the Fe by electrolysis of the perchlorate soln. in a water-cooled cell at a mercury cathode (15 amp., 12 to 14 V, 1 hr.), then removing Hg by pptn. with H_2S in acid soln., and finally measuring the absorption of the molybdenum-blue complex at 650 or 830 mμ by the Hague and Bright procedure (*Brit. Abstr. B I*, 1942, 94). The reference curve is prepared from standard $(NH_4)_2HPO_4$ soln. Errors caused by presence of relatively large amounts of Si and As are eliminated by addition of HF or HBr or both during the final fuming with $HClO_4$. The method can be extended to other suitable high-purity elements or materials. W. J. BAKER

1814. **Behaviour of the condensed phosphates in anion-exchange chromatography.** J. Beukenkamp, W. Rieman III and S. Lindenbaum (*Anal. Chem.*, 1954, **26** [3], 505–512).—Behaviour of the ortho-, pyro-, tri-, trimeta- and tetraphosphate anions on ion-exchange columns is treated theoretically. Consideration is given to ionisation constants of the acids (some of which are calculated from potentiometric titration data), effect of pH and concn. of eluting soln. (buffered Cl′ soln.), and column height. The deductions are confirmed experimentally.
D. A. PANTONY

1815. **Flame-photometric determination of phosphate.** W. A. Dippel, C. E. Bricker and N. H. Furman (*Anal. Chem.*, 1954, **26** [3], 553–556).—A

sample of powdered phosphate rock containing 3·6 to 8·5 mg of P_2O_5 is attacked with a hot mixture of 10 ml of HCl and 5 ml of HNO_3; after baking, SiO_2 is removed by filtration of the diluted residue, and the soln. is passed through a cation-exchange column. To the eluate is added 10 ml of 0·1 per cent. Ca soln., and the mixture is made up to 100 ml. The flame intensity is measured at 422·7 mμ and compared with standards of 100 p.p.m. of $Ca^{\cdot\cdot}$ containing various known amounts of $PO_4^{\cdot\cdot\cdot}$. Mean deviation is given as 1·5 to 2 per cent.

D. A. PANTONY

1816. **Determination of triphosphate and pyrophosphate by isotope dilution.** O. T. Quimby, A. J. Mabis and H. W. Lampe (*Anal. Chem.*, 1954, **26** [4], 661–667).—In a method applicable to commercial triphosphate detergents and Na tripolyphosphates, a known (mg) amount of ^{32}P-tagged pure $Na_5P_3O_{10}$ or $Na_4P_2O_7$ is diluted by inactive $Na_5P_3O_{10}$ or $Na_4P_2O_7$ in the sample (5 to 10 g). The specific activity of the pure $Na_5P_3O_{10}.6H_2O$ or $Na_4P_2O_7.10H_2O$, isolated by 3 to 5 recrystallisations from a (1 + 4) mixture of ethanol and water, is measured on the anhyd. salt. The content of tri- or pyrophosphate in the sample is calculated from the given equations, a correction being made for the systematic error of + 2 per cent. absolute in the triphosphate determination. The wt. ratio of $Na_5P_3O_{10}$ to $Na_4P_2O_7$ in the sample should be $\simeq 4$ to 1 for successful isolation of pure $Na_5P_3O_{10}$ and < 3 to 2 for that of pure $Na_4P_2O_7$. When determining both components, it is necessary to add a known wt. of inactive pure phosphate in at least one of the analyses if not both. Efficiency of purification of the isolated phosphates, limitations of the method and some analyses of commercial detergents are discussed fully. One determination takes 2 to 4 hr., but 1 to 3 days must elapse before results become available. The standard deviations are ± 1 per cent. for $Na_4P_2O_7$ and ± 1·5 per cent. for $Na_5P_3O_{10}$.

W. J. BAKER

1817. **Determination of arsenic and germanium after separation by extraction.** W. Fischer, W. Harre, W. Freese and K.-G. Hackstein (*Angew. Chem.*, 1954, **66** [6], 165–170).—A separation of As^{III} and Ge^{IV} from nearly all other elements can be effected by extraction with CCl_4 from strong HCl. Since As^V is almost insoluble in CCl_4, a mixture of As^{III} and Ge^{IV} can then be separated by oxidising As^{III} to As^V with Br or, preferably, $KClO_3$, and extracting with CCl_4 from HCl soln.; results are satisfactory over the Ge to As range of 10^6 to 10^{-6}.

R. C. MURRAY

1818. **Polarographic analysis for arsenic and antimony.** G. P. Haight, jun. (*Anal. Chem.*, 1954, **26** [3], 593–595).—Pass the gases containing AsH_3 and SbH_3 generated by the action of Zn in 6 *N* H_2SO_4 soln. through small glass tubes containing silica gel impregnated with $AgNO_3$. One g of gel containing 1 per cent. $AgNO_3$ occupying 1 to 2 in. of tube will trap 20 μg of AsH_3 and 50 μg of SbH_3 per min. for 10 min. The $AgNO_3$ darkens to a degree that gives an indication of the approx. amount present. Transfer the silica gel to a small beaker and treat it with 3 ml of soln. containing 1 mole of HCl and 1 mole of tartaric acid per litre on a steam-bath for 10 to 15 min. Pour the soln. into a flask and repeat the extraction twice more. Make up the combined extracts to 10 ml, pass in nitrogen for 10 min. and polarograph. $E_{\frac{1}{2}}$ for Sb^{III} is − 0·138 V *vs.* the S.C.E. and for As^{III} is − 0·403 V *vs.* the S.C.E. The concn. of As and Sb used for polarography were $\simeq 2 \times 10^{-4}$ *M*.

H. F. W. KIRKPATRICK

1819. **Coulometric titrations [of arsenite] with externally-generated chlorine, bromine and iodine.** J. N. Pitts, jun., D. D. DeFord, T. W. Martin and E. A. Schmall (*Anal. Chem.*, 1954, **26** [4], 628–631).—A general-purpose single-arm cell for the external generation of Cl′, Br′ or I′ at currents of 200 to 410 mA is described; generation of I′ is limited to currents $\gtrsim$ 250 mA, with essential addition of 0·1 *M* H_2BO_3 (to neutralise KOH at the cathode) plus 0·5 *M* Na_2SO_4 (in place of strong acids used for Br′ and Cl′ generation). Data on the coulometric titration of arsenite (0·8 to 1·04 milli-equiv. of As) by each of the three externally generated halogens are reported and discussed. A potentiometric end-point is preferred for the titration with Cl′, although amperometric and potentiometric end-points are equally accurate with Br′ and I′. By confining entirely the halide-ion generation within the cylindrical anode (concentric with the tubular cathode), the titrations can be followed potentiometrically with the indicator electrodes placed outside the anode cylinder. The mean error is within 0·1 per cent. The merits and uses of external generation of halogens are discussed.

W. J. BAKER

1820. **Analysis for industry.** [**Gravimetric determination of antimony.**] D. Gibbons (*Ind. Chem.*, 1954, **30**, 183–185).—The pptn. of Sb by means of H_2S, NH_4CNS and thioacetamide, pyrogallol and other organic reagents is reviewed. 45 references.

A. R. PEARSON

1821. **Estimation of trace amounts of bismuth. I. Colorimetric estimation in lead. II. Colorimetric estimation in copper. III. Nephelometric estimation.** B. B. Burnett (*Dissert. Abstr.*, 1954, **14** [2], 231–232).—**I, II.** The preferred method of estimating 10 μg to 10 mg of Bi in 20 g of Pb or 2 g of Cu is by quant. collection, in slightly acid or alkaline soln., as Bi^{III} on SiO_2, followed by elution with dil. H_2SO_4 and measurement of the colour intensity (by the iodide method) at 460 mμ; H_3PO_2 is used to prevent iodide oxidation. Pb should be separated before the gathering process, but Cu is converted to the sol. NH_3 complex and need not be removed. Interference from other elements is negligible. **III.** Nephelometric estimation of $\lesssim$ 0·0·04 p.p.m. of Bi is based on pptn. of cinchonine iodobismuthite by use of a mixture of Tergitol 7 and dextrin to disperse and stabilise the suspension.

W. J. BAKER

1822. **The analysis of combustion products. I. Some improvements in the methods of analysis of peroxides.** Sir A. C. Egerton, A. J. Everett, G. J. Minkoff, S. Rudrakanchana and K. C. Salooja (*Anal. Chim. Acta*, 1954, **10** [5], 422–428).—The stannous chloride method of Barnard and Hargreave (*Brit. Abstr. C*, 1952, 145) is improved by dissolving the organic peroxide (1 milli-equiv.) in 30 per cent. NaOH (20 ml), adding 0·1 *N* $SnCl_2$ (15 ml) in dil. HCl, acidifying after 3 min. with 7·5 *N* H_2SO_4 (25 ml) and titrating with 0·05 *N* I. The whole procedure is conducted under N. In a flow system, H_2O_2 and *tert.*-butyl hydroperoxide are more effectively trapped in aq. NaOH than in acetic acid, and the reaction with $SnCl_2$ is much more rapid. The ferrous thiocyanate method (Kolthoff and Medalia, *Brit. Abstr. C*, 1951, 453) is improved by extracting the colour from the reagent solution, before use, with pentanol. The use of pentanol improves the

stability of the reagent towards atmospheric O and permits the estimation of 0·03 μg of H_2O_2 per ml. In the range 0·3 to 200 μg per ml, hydrogen peroxide is determined as follows. Add 20 ml of conc. H_2SO_4 to 2 ml of 10 per cent. titanous sulphate soln., dilute to 100 ml with water and add dil. H_2O_2 until the bluish colour changes to pale yellow. Mix 1 ml of this reagent with 3 ml of the sample and measure the optical density at 410 mμ. Prepare a calibration curve from known concentrations of H_2O_2.

W. C. JOHNSON

1823. **Determination of sulphur in iron and steel by barium chloride method after chromatographic separation of sulphuric acid.** F. Nydahl (*Anal. Chem.*, 1954, **26** [3], 580–584).—Five g of steel are dissolved in hot conc. HNO_3 + HCl (5 + 1) and the soln. is evaporated to fumes with 50 ml of 70 per cent. $HClO_4$; a similar modified procedure is given for dissolution of iron ores. After cooling, the residue is diluted with 50 ml of H_2O and the insoluble material is removed by filtration. The filtrate is passed through a column of Al_2O_3 prepared for selective ion-exchange by washing with aq. NH_3 and dil. HCl. The eluate and dil. HCl washings are acidified with HCl and treated with 5 drops of 30 per cent. H_2O_2 and 1 ml of acetic acid. A normal determination of SO_4'' by pptn. with Ba^{++} follows, and a blank is allowed for. All interfering ions are claimed to be removed by the ion-exchange procedure, which is discussed in detail. A standard deviation of < 1 per cent. is claimed.

D. A. PANTONY

1824. **Determination of sulphur in iron and steel by the gas evolution method. Corrective and complementary note.** B.-E. Jaboulay (*Chim. Anal.*, 1954, **36** [5], 130–131).—In routine determinations of S in high-speed steels by the thermal evolution method with 3-g samples instead of the usual 10-g ones, there is appreciable action of I on the acidified Zn acetate soln. used to absorb the H_2S. The reaction is negligible, however, if the acetic acid content of the soln. is raised to 4 to 5 g per litre. The I soln. should contain 2·376 g of I plus 8 g of KI per litre, whilst the $Na_2S_2O_3$ soln. should contain 4·644 g per litre. In calculating the S content, a correction coeff. of 1·8 instead of 1·5 should be adopted, as high-speed steels evolve only 54·6 per cent. of their total S during treatment with HCl.

W. J. BAKER

1825. **Determination of sulphur in minerals and slags. Generalisation of the Fincham - Richardson method.** R. Boulin, R. Desguin and E. Jaudon (*Chim. Anal.*, 1954, **36** [5], 123–125).—The Fincham - Richardson method (*J. Iron Steel Inst.*, 1952, **172**, 53) is so modified as to be generally applicable to determinations of S in minerals and basic slags. By placing a porous alundum sample-container (50 mm by 11 mm) on a refractory tray, the combustion products are filtered inside the hot zone and S is integrally liberated. The sample (0·2 to 1 g) is mixed with twice its wt. of pure MgO (to increase porosity and fix P_2O_5), and 50 to 100 mg of pure C (or high carbon Fe of known low S-content) are added to accelerate decomposition of sulphates. Combustion is effected at $\simeq$ 1400° C in an atmosphere of CO_2, and the exit gases are absorbed in an HCl soln. of KI, which is titrated with 0·0125 *N* KIO_3 until the original blue coloration persists. Each determination takes $\simeq$ 10 min.

W. J. BAKER

1826. **Determination of sulphur and barium in organic barium sulphonates.** F. E. Brauns, J. B. Hlava and H. Seiler (*Anal. Chem.*, 1954, **26** [3], 607–608).—Organic barium sulphonate (0·15 to 0·18 g) is dissolved in water (75 ml) and the soln. is passed through a cation-exchange column. The S in the eluate is determined by oxidation with $HClO_4$ - HNO_3, evaporation with HCl and pptn. with $BaCl_2$. The absorbed Ba is extracted with 12 per cent. HCl and pptd. with H_2SO_4. Results for the analysis of 3 barium sulphonates are given.

D. A. PANTONY

1827. **Auxiliary pre-treatment by zinc acetate in sulphide analysis.** R. Pomeroy (*Anal. Chem.*, 1954, **26** [3], 571–572).—Water containing 0·001 to 17 mg of H_2S per litre is treated with 1 ml of 2 *N* Zn acetate per 250 ml, followed by an equal vol. of *N* NaOH or Na_2CO_3. The concn. of S'' can then be determined iodimetrically or colorimetrically on the ppt. Much solid matter and CN' interfere.

D. A. PANTONY

1828. **Spectrophotometric determination of telluric acid.** L. W. Scott and G. W. Leonard, jun. (*Anal. Chem.*, 1954, **26** [3], 445–447).—H_2TeO_4 (65 to 210 mg) is dissolved in water (100 ml) containing 6·6 *M* aq. NH_3 (10 ml), and the absorption of the soln. is measured at 260 mμ (constant temp.). The H_2TeO_4 is determined from a calibration curve, with a precision of $\pm$ 0·8 per cent. Mineral acids, SeO_2, NaOH, acetic acid, $NaAsO_2$ and Na_2HAsO_4 interfere.

D. A. PANTONY

1829. **Chromatographic separation of molybdenum and rhenium.** D. I. Ryabchikov and A. I. Lazarev (*Compt. Rend. Acad. Sci., U.S.S.R.*, 1953, **92** [4], 777–779).—As in acid solution Re^{VII} forms the singly charged ion $ReO_3^{\cdot}$, while Mo^{VI} forms the doubly charged $MoO_2^{\cdot\cdot}$, the latter can be retained on cationites while $ReO_3^{\cdot}$ passes through unadsorbed.

The active groups of cationites in order of decreasing adsorbing power for Mo are: $\cdot SO_2\cdot OH$ < $\cdot CH_2\cdot SO_2\cdot OH$ < $\cdot COOH$ < ($\cdot SO_2\cdot OH$ and $\cdot OH$), Various anionites adsorb MoO_4'' and ReO_4' poorly in alkaline soln., whilst in acid soln., Re is preferentially adsorbed, because of the greater stability of ReO_4'. MoO_4'' can be completely adsorbed by activated Al_2O_3 at pH values between 1 and 6.

R. C. MURRAY

1830. **Polarographic determination of sexavalent uranium.** V. B. Vouk, M. Branica and O. A. Weber (*Arhiv. Kem.*, 1953, **25** [4], 225–229).—A polarographic method is proposed for the determination of small amounts of U^{VI}. An aq. soln. of salicylic acid (1·6 g per litre), H_2SO_4 (0·4 per cent. v/v) and thymol (0·009 per cent.) is used as supporting electrolyte. The concn. of U in the soln. for polarographic measurement should be between 5 and 50 μg per ml, and all dissolved O must be completely removed from the soln. before analysis. Total amounts of 10 μg of U per sample can be determined with a standard error of $\pm$ 0·8 μg.

D. BAILEY

1831. **Use of a cation-exchange resin for total anion analysis of aqueous solutions containing uranyl ion.** H. O. Day, jun., J. S. Gill, E. V. Jones and W. L. Marshall (*Anal. Chem.*, 1954, **26** [3], 611–612).—Through a cation-exchange column prepared with 3 *N* H_2SO_4 is passed 35 ml of a soln. of 1 g of UO_3 in a definite quantity of the acid anion under consideration. The eluate is made up to 100 ml and titrated with standard NaOH. SO_4'', NO_3', Cl', ClO_4' and Cr_2O_7'' are determined in this manner with an error of $\pm$ 0·15 per cent.

D. A. PANTONY

1832. **Quantitative separation of three halogens.** E. Kahane and M. Kahane (*Bull. Soc. Chim. France*, 1954, **21** [3], 396–400).—The quant. separation of Br′ from Cl′ by oxidation with HNO_3 is investigated. In the hot or cold, some Cl′ is always oxidised at a concn. of HNO_3 high enough to complete the oxidation of Br′. The rate of oxidation in the cold is reduced by stirring, owing to reduction in the concn. of dissolved N oxides; it is increased by addition of $NaNO_2$. I′, Br′ and Cl′ can be determined if 5 ml of solution, 1 ml of 30 per cent. $NaNO_2$ and 1 ml of glacial acetic acid are introduced into a bubbler connected in series with 4 bubblers containing 10 ml of 6·6 *N* HNO_3, one bubbler containing 10 ml of 10 per cent. Na_2SO_3 and one bubbler containing 10 ml of 10 per cent. NaOH. Passage of air for 45 min. sweeps all the I into the NaOH. One ml of 30 per cent. $NaNO_2$ and 8 ml of HNO_3 are then added to the first bubbler. Passage of air for 45 minutes sweeps all the Br into the Na_2SO_3, Cl being retained in the first five bubblers. See also *Anal Abstr.*, 1954, **1**, 292.
A. B. DENSHAM

1833. **Automatic coulometric titration with electrolytically generated silver ion. Determination of chloride, bromide and iodide ions.** J. J. Lingane (*Anal. Chem.*, 1954, **26** [4], 622–626).—Cl′, Br′ and I′ are titrated coulometrically with Ag⁺ generated electrolytically with const. current (5 to 100 mA) at a silver anode immersed directly in the sample solution, the equivalence-point being detected potentiometrically with a silver indicator electrode. A Beckman titrator adapted to effect these titratiins automatically within 1 to 5 min. is described. The preferred supporting electrolyte ($\simeq$ 50 ml) is aq. 0·5 *M* $HClO_4$, or 0·4 *M* $NaNO_3$ plus 0·05 *M* $HClO_4$, with addition of 80 per cent. ethanol or 75 per cent. acetone if the concn. of halide ion is $< 0{\cdot}5$ mg per 50 ml. Corrections for end-point and incremental errors are made; for ion concn. of 0·2 to 10 mg per 50 ml, the mean error is $\pm$ 0·005 mg. The method is regarded as of more general application.
W. J. BAKER

1834. **Volumetric determination of fluorine.** G. Brunisholz and J. Michod (*Helv. Chim. Acta*, 1954, **37** [2], 598–602).—The sample (1 to 10 mg of F in a max. vol. of 50 ml) is titrated with 0·1 *N* or 0·2 *N* NaOH (free from Na_2CO_3) until bromothymol blue is permanently blue and then with 0·1 *N* HCl until it is yellow (excess must be avoided). After addition of an equal vol. of methanol and sufficient murexide soln. to give a not too intense rose-violet colour, the soln. is titrated slowly with 0·05 *N* $CeCl_3$ (standardised by pptn. as oxalate and weighing as CeO_2). The colour of the soln. under examination is compared with that of a standard soln. of $CoCl_2$ to which it gradually approaches. At the end-point it passes from rose-violet to rose-orange and a distinct difference is observed in the colour of the two soln. Allowance must be made for the quantity of $Ce^{\cdots}$ required to operate the indicator.
H. WREN

1835. **Determination of fluorine in inorganic phosphates.** K. W. Gerritsma and J. C. Frederiks (*Chem. Weekbl.*, 1954, **50** [20], 356–358).—The fluorine content of inorganic phosphates is determined colorimetrically by means of zirconium alizarin dye after distillation as fluorosilicic acid. The interference of HCl is eliminated by neutralising the distillate. Amounts from 7 p.p.m. of F can be determined with an accuracy of $\pm$ 4 per cent. In 40 samples of phosphates used as food additives, the F content varied from < 7 to 2300 p.p.m. The fluorine can also be qualitatively detected as typical crystals of barium fluorosilicate by means of a microchemical method.
A. STORFER

1836. **Accurate and rapid method for the determination of fluorine in glasses and raw materials.** W. Vogel (*Silikattech.*, 1953, **4**, 483–485).—In combination with a $HClO_4$-distillation method F′ is determined by a method based on Flatt's potentiometric titration of H_2SiF_6 with U salts. The method is equally suitable for fluorides, complex fluorosalts, etc., even in the presence of B, Be, Ti, Al and Th. The distillation apparatus of Balczo - Kaufmann (*Mikrochem. Mikrochim. Acta*, 1951, **38**, 237) is used, in which the sample is decomposed by 60 per cent. $HClO_4$ at a const. temp. of 142° C (amyl acetate as the bath liquid). The F′ is distilled as H_2SiF_6. The distillate is neutralised with 2 *N* NaOH soln. A special correction is applied for the F losses, according to Richter (*Z. anal. Chem.*, 1942, **124**, 161). The pH must be kept const. at 3·4 during the titration. K sulphanilate (in 0·5 *N* soln.) is used as buffer substance. The titration is done in a CO_2 atmosphere with an amplifying electrical device. The accuracy of the method is $\pm$ 0·1 to 0·2 per cent. for 20 to 250 mg of F and 1 to 1·5 per cent. for 700 to 800 mg of F. The max. time required for the smallest quantities of F is 35 min. compared with 6 to 12 hr. if pptd. as PbBrF.
CHEM. ABSTR.

1837. **Colorimetric determination of fluoride.** M. L. Nichols and A. C. Condo, jun. (*Anal. Chem.*, 1954, **26** [4], 703–707).—Concn. of < 6 p.p.m. of F in water can be determined accurately by utilising the bleaching action of F on the Fe^{III} complex of 5-phenylsalicylic acid (in ethanol) or of resacetophenone (in 10 per cent. acetone), both complexes being stable over long periods. At pH 2 to 3 and with 5 to 10 p.p.m. of Fe^{III}, 1 ml of reagent and a 0·1 *M* buffer, a 4 per cent. change in transmission (measured at 575 or 465 mμ with a photometer having 2-cm cells and 30-mμ band filters) corresponds to 1 p.p.m. of F. More than 5 p.p.m. of Al interfere, as do ions that form stable complexes with Fe^{III} (*e.g.*, citrates, tartrates). A number of other organo-ferric complexes studied were unsuitable for the method.
W. J. BAKER

1838. **The polarographic determination of fluoride. I. Basic principles of the method: application to the cathode-ray polarograph.** B. J. MacNulty, G. F. Reynolds and E. A. Terry (*Analyst*, 1954, **79**, 190–198).—A sensitive method for determining F′ polarographically is described. It is developed from an observation by Willard *et al.* (*Brit. Abstr. C*, 1951, 164) of the interference of F′ in their method for the polarographic determination of Al with Solochrome violet RS. The polarographic step given by the reduction of the Al - dye complex is depressed by F′, and the depression is linearly related to the amount of F′ down to 0·001 μg per ml. For sub-microgram amounts of F′, a cathode-ray polarograph is required.
A. O. JONES

1839. **Determination of fluoride ion using a monohydroxy azo-dye - thorium lake.** J. L. Lambert (*Anal. Chem.*, 1954, **26** [3], 558–560).—A filter-paper is treated in a standard manner with a 0·2 per cent. soln. of amaranth and, after drying, the paper is soaked in 1 per cent. aq. $Th(NO_3)_4$. After several water washings, the moist paper is placed in a dish containing 20 ml of F′ (1 to 40

p.p.m.) for 5 min., and the absorption of the resulting soln. of free amaranth dye is measured at 521 mμ, at which the relationship between concn. of F′ and absorption is linear. No precision data are given; SO_4'', HPO_4'', HCO_3', H^+, OH', Al^{+++} and Fe^{+++} interfere seriously. D. A. PANTONY

1840. **Mercurimetric determination of chlorides.** J. Ungar (*Chem. & Ind.*, 1954, [16], 453–454).—Diphenylcarbazone was used as indicator, the accuracy of the procedure being greatest when the pH was ≯ 8·4. Variation in strength of titrating solution, selection of solvent for the indicator and interference caused by common ions were also examined. An absorptiometric calibration curve for NaCl concn. up to 1 p.p.m. is shown.
M. TADMAN

1841. **Titration of iodide with electrolytically generated silver ion.** R. L. Kowalkowski, J. H. Kennedy and P. S. Farrington (*Anal. Chem.*, 1954, **26** [4], 626–628).—Secondary coulometric titration of I′ is effected automatically by means of electrolytically generated Ag^+ and the amperometric end-point is detected by means of two small silver indicator electrodes at an impressed p.d. of ≃ 230 mV. Control of indicator current and influence of side reactions (reduction of H^+, etc.) and supporting electrolyte are discussed. In acetate buffer soln. (pH 5·5), the mean error is ± 0·1 per cent. for I′ concn. of 0·7 to 23 mg per 75 ml and ± 0·6 per cent. for concn. < 0·5 mg. An alternative coulometric titration of ≃ 3 mg of I′ in 0·02 *M* H_2SO_4, involving determination of approx. and final end-points, is also described; the mean error is ± 0·1 per cent.
W. J. BAKER

1842. **Determination of periodic acid in highly coloured solutions.** M. A. Smith and B. R. Willeford, jun. (*Anal. Chem.*, 1954, **26** [4], 751–753).—The consumption of periodate by flavonol, quercetin and similar compounds during their oxidation with HIO_4 is determined by passing the highly coloured reaction mixture through an ion-exchange resin column (Amberlite IRA-400 converted to acetate) so as to obtain 99 ± 0·6 per cent. recovery of periodate, which is eluted from the column (20 cm by 8 or 12 mm) with aq. 5 per cent. NaOH at a rate of 1 to 2 ml per min. to give a total recovery of ≃ 99·3 per cent. The data obtained show consumptions of ≃ 1 mol. of periodate per mol. of flavonol during a 20 to 60-hr. reaction, and of ≃ 2 mol. of periodate per mol. of quercetin within ≃ 15 to 30 min., rising to 3·5 mol. after a 94-hr. reaction.
W. J. BAKER

1843. **Studies on substitutes for the Zimmermann - Reinhardt reagent [for iron].** K. M. Somasundaram (*J. Sci. Ind. Res., B, India*, 1954, **13** [1], 35–37).—For the Zimmermann - Reinhardt reagent ($MnSO_4$, H_2SO_4 and H_3PO_4), which is used in the titration of Fe^{++} with $KMnO_4$ in presence of HCl, Na acetate and borax were found to be excellent substitutes. The end-point in presence of Na acetate is indicated by a change from yellow to orange, but if the yellow colour is first discharged by adding Na_3PO_4, the titration can be carried out in the ordinary way. Sodium acetate (2 g) and Na_3PO_4 (2 g) added to each 20 ml of 0·1 *N* Fe^{++} soln. and 20 ml of 5 *N* HCl replace 40 ml of Zimmermann - Reinhardt reagent.
J. M. JACOBS

1844. **Iodimetric determination of ferricyanide in the presence of copper, silver, cadmium and zinc.** M. Kohn (*Anal. Chim. Acta*, 1954, **10** [5], 405–407).—Freshly pptd. ferricyanides of Cu, Cd, Ag and Zn can be converted quant. to ferrocyanides by adding potassium ferrocyanide to their aq. suspensions. If the metallic ferrocyanide is coagulated with an electrolyte and filtered off, the ferricyanide can be titrated iodimetrically in the filtrate. The Zn salt need not be filtered off, and filtration can be avoided if silver ferricyanide is decomposed with KI instead of with ferrocyanide.
W. C. JOHNSON

1845. **Possible loss of iron during sodium carbonate fusion of silicates and rocks.** H. R. Shell (*Anal. Chem.*, 1954, **26** [3], 591–593).—Losses of Fe on ignition of silicate rocks with Na_2CO_3 in platinum vessels are examined. Determination of Fe_2O_3 (colour with *o*-phenanthroline) on HCl extractions of the Pt shows that fusion of the sample over a gas burner or in an electric muffle furnace causes no appreciable loss, but fusion followed by SiO_2 ignition over a flame causes serious losses (up to 80 per cent., depending on the silicate), which may be avoided by carrying out the ignition of SiO_2 in an electric muffle furnace.
D. A. PANTONY

1846. **Microscopic identification of wustite in presence of other oxides of iron.** R. G. Wells (*Anal. Chem.*, 1954, **26** [4], 715–717).—Microscopic methods of distinguishing between wustite (FeO), haematite and magnetite in partly reduced or sintered iron ores are described. Although wustite closely resembles magnetite (both are black and isotropic), it is easily scratched with a steel needle, has a Knoop indentation hardness of 155 (magnetite 361) and, unlike the other oxides, is strongly etched within 1 to 2 min. by a saturated soln. of $SnCl_2$ in ethanol. Haematite has a greater reflectivity than wustite or magnetite is anisotropic, and yields a red non-magnetic powder when scratched.
W. J. BAKER

1847. **Determination of cyanides and iron in liquors and solids, and of cyanides and sulphur in spent oxide.** A. Adelsberger (*BrennstChemie*, 1954, **35** [5–6], 75–79).—The sample, *e.g.*, a gas condensate or washing liquor, is shaken for 40 min. at 130° C in a sheathed porcelain or enamelled iron vessel (described) with a 3 to 4-fold excess of ammonium polysulphide, whereby all ^-CN present as cyanide, ferrocyanide, etc., goes into solution as CNS′ and is titrated, after filtration, with 0·1 *N* $AgNO_3$. The FeS on the filter is washed and ignited to Fe_2O_3. In the examination of spent oxide the total ^-CN and free S are simultaneously extracted by aq. $(NH_4)_2S$ (monosulphide) at 130° C. FeS is filtered off and washed with aq. NH_3. The filtrate is boiled to expel NH_3 and H_2S and to ppt. S in solution as polysulphide and thiosulphate, the pptn. being completed by dropwise addition of 5 *N* H_2SO_4. The S is filtered off and weighed. The filtrate, decolorised if necessary by activated C, is titrated with 0·1 *N* $AgNO_3$, when the total ^-CN is calculated. By treating the spent oxide with aq. $Fe_2(SO_4)_3$, ferrocyanide can be pptd. and filtered off, along with free S, for separate determination as above, whilst CN′ and CNS′ are determined in the filtrate.
A. R. PEARSON

1848. **Spectrophotometric determination of cobalt with 2-nitroso-1-naphthol-4-sulphonic acid.** W. M. Wise and W. W. Brandt (*Anal. Chem.*, 1954, **26** [4], 693–697).—Concn. of 10^{-5} to 10^{-4} *M* (0·6 to 6 p.p.m.) of Co^{++} are determined accurately by measuring the colour intensity at 525 mμ of the water-sol. red complex formed by reaction of Co^{II} with 0·01 *M* 2-nitroso-1-naphthol-4-sulphonic acid

at pH 7. Large amounts of Ni^{++}, Fe^{+++} and Cu^{++} should be removed completely, but concn. of $< 0{\cdot}001$ *M* of Cu^{++} or Fe^{+++} can be sequestered as citrate complexes. If the Ni^{II} to Co^{II} ratio is $\gtrsim 20$ the absorption can be measured at 560 mμ without serious error. The system is stable, independent of reagent concn. and obeys Beer's law for concn. of Co between 4×10^{-6} and $1{\cdot}25 \times 10^{-4}$ *M*.

W. J. BAKER

1849. **Potentiometric determination of cobalt by dead-stop titration.** H. Fritzsche (*BrennstChemie*, 1954, **35** [3–4], 49–51).—Co^{++} in aq. NH_4Cl - aq. NH_3 at 20° C is oxidised to Co^{+++} by excess of 0·1 *M* $K_3Fe(CN)_6$, which is back-titrated with 0·1 *M* $Co(NO_3)_2$ while mechanically stirred. The p.d. between the platinum electrodes is read from a sensitive millivoltmeter. Addition of citric acid is unnecessary as Fe, Al and Th are pptd. from the soln. Ni^{++}, Ca^{++}, Mg^{++} and F' do not affect the titration. See *Brit. Abstr. C*, 1950, 321.

A. R. PEARSON

1850. **Separation of [tervalent] rhodium from [quadrivalent] iridium.** G. H. Ayres and C. M. Maddin (*Anal. Chem.*, 1954, **26** [4], 671–674).—The efficiency of separation of Rh^{III} from Ir^{IV} by selective reduction of Rh^{III} to Rh with $TiCl_3$ at 100° C is examined by spectrographic and spectrophotometric methods. By using the porous-cup electrode technique with a high-voltage spark and the line-pairs Rh 3434·9 - Co 3455·2 and Ir 2829·7 - Co 3455·2, it is possible to detect a min. of 0·5 p.p.m. of Rh and 50 p.p.m. of Ir to within $\simeq$ 3 per cent.; lower concn. of Ir are determined by a modification of the Ayres spectrophotometric method (*Brit. Abstr. C*, 1951, 131). For Rh - Ir ratios between 10 to 1 and 1 to 10, the separation efficiency is 99·7 per cent. for each element. The wt. of Rh failing to ppt. (0·02 to 0·5 mg on 10 to 100-mg samples) and the wt. of co-pptd. Ir (0·02 to 0·3 mg) are independent of the wt. of the other element present. The small compensating errors make unnecessary a second pptn. unless the highest accuracy is required.

W. J. BAKER

1851. **Separation of palladium from lead and the colorimetric determination of palladium with potassium iodide.** J. G. Fraser, F. E. Beamish and W. A. E. McBryde (*Anal. Chem.*, 1954, **26** [3], 495–498).—A clean assay lead button (15 to 30 g) containing 1 to 1000 μg of Pd is dissolved in dil. (1 + 2) HNO_3 (90 ml), the soln. is evaporated to dryness and the residue is taken up in dil. (1 + 500, slightly stronger if Cu^{++} is present) HNO_3 (100 ml), any Fe^{+++} being complexed by addition of 0·5 g of Versene. One per cent. aq. dimethylglyoxime (Na salt) is added, and after 1 hr., the soln. is extracted with 4 portions of $CHCl_3$ (100 ml in all). After evaporation to dryness, the residue is decomposed with hot (1 + 1) mixture of conc. H_2SO_4 and conc. HNO_3 (12 ml) and after cooling is diluted and filtered; slightly ammoniacal 50 per cent. KI reagent (1 ml per 100 μg of Pd) is then added. After addition of 5 ml of 0·6 per cent. Na_2SO_3, the soln. is made up to 100 ml, and the Pd, as PdI_4'', is determined spectrophotometrically at 408 mμ. Alternatively, a lead button containing 5 mg of Pd is dissolved in dil. (1 + 2) HNO_3 (90 ml), the soln. being evaporated to dryness after dilution and filtration. The residue is dissolved in dil. (1 + 500) HNO_3 (100 ml) and the hot soln. is filtered; to it is added saturated aq. salicylaldoxime (10 ml). After digestion, the ppt. is filtered on close paper, ignited in porcelain, reduced in H and cooled under N. The residue is dissolved in, and evaporated to dryness with *aqua regia* thrice and then with HCl thrice, the final residue being dissolved in dil. (1 + 32) HCl (8 ml). The hot soln. is diluted with 25 ml of H_2O, filtered and diluted to 100 ml; the Pd is re-precipitated with salicylaldoxime and the complex is dried at 115° C.

D. A. PANTONY

1852. **Study of osmium and ruthenium distillations.** A. D. Westland and F. E. Beamish (*Anal. Chem.*, 1954, **26** [4], 739–741).—Methods of separating macro- and micro-amounts of Os and Ru (in assay beads, etc.) and of determining each element gravimetrically or colorimetrically are investigated. From soln. containing both metals, Os, free from Ru, is completely distilled by using $HClO_4$ as oxidant and collecting the distillate in 48 per cent. aq. HBr. By treating the sample soln. so as to form the tetroxides, passing the vapours into 3 per cent. H_2O_2 at 0° C and boiling subsequently with 30 per cent. H_2O_2 (40 ml) plus conc. H_2SO_4 (5 ml) for $\simeq$ 30 min., Os is completely separated from Ru (retained in the peroxide soln.) and can be collected finally in aq. HBr. For samples containing > 15 mg of both metals, extra H_2O_2 is needed in the distillation flask to keep Ru in the reduced state; Ru is recovered by distillation with additional conc. H_2SO_4 (10 ml) and excess of 10 per cent. aq. $NaBrO_3$ according to the method of Thiers *et al.* (*Anal. Chem.*, 1948, **20**, 831). The Ru and Os contents of the two distillates are determined gravimetrically with thionalide for amounts of metal > $\simeq$ 3 mg, or spectrophotometrically as the thiourea complexes, for μg amounts, by slight modifications of the standard procedures of Ayres *et al.* (*Anal. Chem.* 1950, **22**, 1277) and Allan *et al.* (*Anal. Chem.*, 1952, **24**, 1569). The gravimetric error is $\simeq \pm 0{\cdot}1$ mg, and the error in the colorimetric method, which is applicable in presence of Pt, Pd, Rh or Ir, is $\simeq \pm 5$ μg.

W. J. BAKER

1853. **Absorptiometric determination of iridium and rhodium.** A. D. Maynes and W. A. E. McBryde (*Analyst*, 1954, **79**, 230–238).—Procedures for the absorptiometric determination of Ir and Rh have been devised. Ir in the form of sulphate is oxidised by $Ce(SO_4)_2$ to produce a red solution, the optical density of which at 510 mμ is linearly related to the concn. The $SnCl_2$ procedure for determination of Rh (Sandell, "*Colorimetric Determination of Traces of Metals*," Second Edition, Interscience Publishers Inc., New York and London, 1950, pp. 523–525) was examined with special reference to the effect of Ir. A procedure was then developed for application of these methods in sequence to determinations of Ir and Rh (in that order) in the same solution without previous separation.

A. O. JONES

1854. **On the thermogravimetry of analytical precipitates. LXIII. Determination of platinum.** P. Champ, P. Fauconnier and C. Duval (*Anal. Chim. Acta*, 1954, **10** [5], 443–447).—Thermogravimetric curves are determined for the various forms in which Pt is pptd. in its gravimetric determination. For each method considered the precipitant, the form in which the Pt can be weighed and the temp. range in which the weighed form is produced are stated as follows: (*i*) Mg, Pt, 100° to 600° C, (*ii*) H_2S, Pt, > 421° C, (*iii*) NH_4^+, $[(NH_4)_2PtCl_6 \rightarrow]Pt$, 407° to 538° C, (*iv*) K^+, K_2PtCl_6, 54° to 270° C, (*v*) Rb^+, Rb_2PtCl_6, 70° to 674° C, (*vi*) Cs^+, Cs_2PtCl_6, 200° to 409° C, (*vii*) Tl^+, Tl_2PtCl_6, 65° to 155° C, (*viii*) thioformamide, Pt, 407° to 625° C, (*ix*)

thiophenol (**I**), $(C_6H_5S)_2Pt$, 230° to 300° C, (*x*) thiophenol, Pt, > 455° C, (*xi*) thiosemicarbazide, Pt, > 437° C, (*xii*) tetraphenylarsonium bromide (**II**), Pt, > 550° C, and (*xiii*) α-furildioxime, Pt, > 370°C. Pt has previously been weighed only as Pt when pptd. with **I** (Currah *et al.*, *Brit. Abstr. C*, 1946, 155). It has been proposed (Bode, *Brit. Abstr. C*, 1952, 383) to ppt. Pt with **II** and to weigh the $[PtCl_6][(C_6H_5)_4As]_2$, but the existence of no stable compound with this reagent is indicated by a thermogravimetric curve, and results are not consistent. W. C. JOHNSON

1855. **Chemico-physical researches on coals, cokes and graphites. VI. Reflectivity and reflection anisotropy of vitrainites.** E. Wege (*BrennstChemie*, 1954, **35** [3–4], 33–41).—Reflectivities (*R* per cent.) from coals of fixed carbon content (*F*) (52 to 98 per cent.) have been determined at 530 mμ in air and at 589 mμ oil-immersed. The results for the two colours correlate closely. The locus of the *R* values is a band rising continuously with *F* and especially rapidly in the anthracite region. The anisotropy (*A*) of the reflectivity rises slightly with *R* from *R* = 8 to 13 per cent., but is much higher for the anthracites, where *R* = 14 to 16 per cent. Neither *R* nor *A* is precise enough to be used to fix the rank of a coal. A. R. PEARSON

See also Abstracts 1755, 1756, 1757, 1993, 1994, 1995, 2009.

3.—ORGANIC ANALYSIS

1856. **Semimicro system of qualitative and quantitative elementary analysis.** E. H. Swift and C. Niemann (*Anal. Chem.*, 1954, **26** [3], 538–543).—A semimicro system of qual. and quant. analysis of 32 elements in organic compounds is described. A 20 to 40-mg sample is fused with Na_2O_2 in a Parr bomb. Treatment of the fused mass with water results in a residue consisting of basic elements forming insol. oxides and carbonates. The soln. contains the amphoteric and acidic elements. Both residue and soln. are systematically analysed. The limit of detection for any element is 1 per cent. and the accuracy is ≯ ± 0·3 mg. Representative analyses for several pure compounds and mixtures are given. G. P. COOK

1857. **Avoidance of the use of lead dioxide in the micro-determination of carbon and hydrogen.** E. Abramson with A. Brochet (*Bull. Soc. Chim. France*, 1954, **21** [3], 367–369).—PbO_2 does not completely absorb N oxides in the micro-combustion of compounds rich in N. Determinations of C and H are satisfactory if the PbO_2 is replaced by Ag on pumice, and a tube containing Al_2O_3 impregnated with diphenylamine in H_2SO_4, followed by one containing $Mg(ClO_4)_2$, is placed between the absorbers for H_2O and CO_2. The reagent is prepared by stirring Al_2O_3 into 1 per cent. w/v diphenylamine in H_2SO_4, and pressing the mixture on a porous plate. A. B. DENSHAM

1858. **Technique for the determination of tritium in organic compounds containing tritium.** R. Viallard, M. Corval, B. Dreyfus-Alain, M. Grenon and J. Herrmann (*Chim. Anal.*, 1954, **36** [4], 102–104).—A semimicro combustion train adapted for isotopic determinations is described. A pre-oxidation tube and series of absorbers serve for purification of oxygen; the combustion tube is followed by a trap immersed in CO_2 snow to condense the water of combustion, and this is followed by a control trap of anhydrone. Finally there are absorbers for determination of CO_2 and ^{14}C if required. The condensed water is transferred to a reduction apparatus consisting essentially of a column of magnesium turnings heated to 480° C; the resulting hydrogen is collected and its β-particle emission determined by Geiger counters. The method is sensitive to $\simeq 10^{-14}$ g-atom amounts of tritium per mole and usually requires only 0·01 to 0·02 g of sample. H. F. W. KIRKPATRICK

1859. **Direct determination of oxygen in organic substances. Modified Schütze method.** I. J. Oita and H. S. Conway (*Anal. Chem.*, 1954, **26** [3], 600–602).—Two modifications are suggested to overcome errors in the Schütze method due (*i*) to the high C temp required for complete conversion of O to CO and (*ii*) to the errors caused by S. Instead of plain C at 1120° C, 50 per cent. platinised C at 900° C is used, and in order to decompose CS_2 and carbonyl sulphide the gas stream emerging from the platinised C is passed over Cu at 900° C and then through Ascarite. The liquid-nitrogen trap of the original method is discarded. By this modified method, blank determinations are usually negligible and both accuracy and repeatability are within 0·15 per cent. for O. G. P. COOK

1860. **Quantitative determination of organic nitrogen by flame spectroscopy.** M. Honma and C. L. Smith (*Anal. Chem.*, 1954, **26** [3], 458–462).—Sixty-five nitrogenous compounds were studied and each one yielded a definite cyanogen spectrum in the inner cone of a flame; the cyanogen band at 388·3 mμ was used as the analysis line and the CH· band at 389 mμ was used as the internal-standard line. The log intensity ratios of these line pairs plotted against log per cent. of N gave satisfactory working graphs. The optimum working range for N was 0·1 to 15 per cent., and the limit of sensitivity was 0·8 mg of N in 3 ml of soln. Reproducibility data from the same compound showed a standard deviation of $\simeq \pm 2$ per cent. of the mean. G. P. COOK

1861. **Micro-determination of nitrogen in volatile compounds.** W. J. Schenck (*Anal. Chem.*, 1954, **26** [4], 788).—The volatile compound is weighed in a sealed capillary, which is lightly scored with a glass cutter before insertion into the combustion apparatus. After the initial sweeping out process, the capillary is broken by a device from outside, and the analysis is completed in the usual way. A. J. MEE

1862. **Determination of organic halogen with sodium diphenyl reagent.** L. M. Liggett (*Anal. Chem.*, 1954, **26** [4], 748–750).—The organic halide is decomposed by the action of Na or Li diphenyl in 1 : 2-dimethoxyethane soln. Following extraction with water, the halide ion is determined by the Volhard method or other conventional means. A method for the preparation of the diphenyl reagent, which is stable for 1 to 2 months, is also described. The accuracy and precision are 2 and 1·5 per cent., respectively. No Cl or Br compounds have been found for which the reagent is ineffective. G. P. COOK

1863. **Quantitative combustion of fluorine-containing organic compounds.** R. Wickbold (*Angew. Chem.*, 1954, **66** [6], 173–175).—The sample for analysis is volatilised by heating in a stream of O in a quartz

tube, the mixture with O is burned and the combustion product is absorbed. F is converted to HF, which is absorbed in *N* NaOH. It is also possible to determine small quantities of F in liquids by combining the combustion device with an atomiser so that relatively large amounts of material can be handled. R. C. MURRAY

1864. **Use of apparent dissociation constants in qualitative organic analysis.** T. V. Parke and W. W. Davis (*Anal. Chem.*, 1954, **26** [4], 642–645).—Procedures are described for obtaining and interpreting potentiometric titration data in the form of H^+ binding *vs.* pH curves. The determination of dissociation constants for acidic and basic groups by this procedure leads to a means of identification of many organic groups. The methods are demonstrated by application to α-amino-β-mercapto*iso*valeric acid, glycine, *p*-aminophenol, Na acetate, adipic acid and aureomycin. A list of the p*Ka* values of dissociating groups is also included. G. P. COOK

1865. **Use of iodine monochloride in the Kurt Meyer titration.** A. Gero (*Anal. Chem.*, 1954, **26** [3], 609).—The Kurt Meyer titration is improved by substituting ICl for Br. To eliminate the catalytic effect of the HCl formed in the addition, $NaHCO_3$ is added in amounts approx. equivalent to the amount of enol present, and being insol. in methanol, it does not interfere with the equilibrium. The limit of accuracy of this modification is $\simeq 10^{-6}$ *M* of enol. G. P. COOK

1866. **Semimicro-determination of hydroxyl group in long-chain alcohols.** R. T. Blickenstaff, J. R. Schaeffer and G. G. Kathman (*Anal. Chem.*, 1954, **26** [4], 746–748).—The alcohol is sulphated with chlorosulphonic acid, the mixture is adjusted to pH 8 and the Na alkyl sulphate is determined by titration with cetyltrimethylammonium bromide and use of methylene blue as indicator. As short-chain alcohols (C_1 to C_4) give alkyl sulphates that are not titrated under the conditions described, they are not expected to interfere. Mercaptans, olefines and cholesterol interfere. The standard deviation is ± 0·079 per cent. G. P. COOK

1867. **Detection, estimation and removal of impurities in fluorocarbon liquids.** D. Grafstein (*Anal. Chem.*, 1954, **26** [3], 523–525).—Treatment of liquid fluorocarbons with solid KOH at 135° C eliminates traces of ethenoid and hydrogen-containing compounds having high absorptions in the u.v. The purification is followed by means of spectrophotometric measurements at 226 mμ. D. A. PANTONY

1868. **Colorimetric determination of glycerol.** H. D. Reese and M. B. Williams (*Anal. Chem.*, 1954, **26** [3], 568–570).—The method is based on the reduction of $K_2Cr_2O_7$ by glycerol in H_2SO_4 soln., the $Cr_2(SO_4)_3$ forming a complex with *s*-diphenylcarbazide whose colour is measured at 540 mμ. The glycerol content of the sample is calculated from this measurement. Accuracy over a glycerol range of 1 to 13,000 μg per ml is good. G. P. COOK

1869. **Spectrophotometric modification of the cuprous oxide method for determining sugars.** M. E. Hobbs and F. L. Layton (*Anal. Chem.*, 1954, **26** [3], 585–586).—After being filtered and washed, the Cu_2O ppt. is dissolved in 4 *M* HNO_3 (75 ml at ≃ 75° C), and the soln. is diluted to 200 ml. A 10-ml aliquot is mixed with 10 ml of *M* ethylenediamine and is diluted to 50 ml. The absorption is measured at 550 mμ, the Cu is measured by reference to a calibration graph and the sugar content is calculated from this. The precision of this modification, for absorbances > 0·1 is between 0·5 and 1·0 per cent. of the Cu present. G. P. COOK

1870. **Micro-determination of acetaldehyde as its 2 : 4-dinitrophenylhydrazone.** G. R. A. Johnson and G. Scholes (*Analyst*, 1954, **79**, 217–219).—A colorimetric method for determination of acetaldehyde is described. The 2:4-dinitrophenylhydrazone is formed in aq. soln. and is extracted quantitatively by CCl_4. Addition of ethanolic NaOH to this extract produces a red colour, which is measured absorptiometrically. Amounts as small as 5 μg per 20 ml of sample can be estimated. With > 200 μg per 20 ml, the calibration graph is not linear. The method is applicable in presence of pyruvic acid, because the interfering hydrazone can be removed by extraction with aq. Na_2CO_3. A. O. JONES

1871. **Reaction of acetaldehyde with 5:5-dimethyl*cyclo*hexane-1:3-dione.** G. W. Gaffney, W. A. Williams and H. McKennis, jun. (*Anal. Chem.*, 1954, **26** [3], 588–589).—The practicability of separating aldehydes and ketones by aeration procedure was investigated, the main reference being the removal of acetaldehyde by quant. absorption in aq. soln. of dimedone (5:5-dimethyl-*cyclo*hexane-1:3-dione). The results showed that absorption is quant. when the pH of the dimedone soln. is between 5 and 6 and that the absorption is independent of the type of alkaline reagent used. By having a high concn. of dimedone in the absorbant, pH shift was decreased, owing to the buffering effect. G. P. COOK

1872. **Spectrophotometric determination of glyoxal.** J. M. Dechary, E. Kun and H. C. Pitot (*Anal. Chem.*, 1954, **26** [3], 449–452).—Two methods are described, the first being suitable for pure α-dicarbonyl compounds and the second for the determination of glyoxal in biochemical material. Both methods are based on the colour reaction with 2:3-diaminophenazine, the excess of colour reagent being removed by tetra-azotisation and reduction of the tetrazonium salt with H_3PO_2. The first method is carried out in 15·8 *N* H_2SO_4, with prolonged heating, and the second in 4·3 *N* acetic acid with a short heating time. The wavelength used for the determination depends on what α-dicarbonyl compound is required; that for glyoxal is 600 mμ. By this procedure 0·01 to 0·2 μ*M* concn. of glyoxal were determined. G. P. COOK

1873. **A simple chromatographic method for the determination of the saturated straight-chain fatty acids C_{10} to C_{24}.** H. J. Nijkamp (*Anal. Chim. Acta*, 1954, **10** [5], 448–458).—The naturally occurring saturated straight-chain fatty acids C_{10} to C_{24} are separated by partition chromatography and a technique similar to that already used for the lower acids (Nijkamp, *Brit. Abstr. C*, 1952, 199). Silica gel is impregnated with aq. methanol, coloured with bromothymol blue and treated with sufficient aq. NH_3 to produce a bluish-green colour. The acids are dissolved in 2:2:4-trimethylpentane saturated with 95 per cent. methanol the same solvent being used for elution. The separated bands of the acids are observed through the colour change of the indicator. The acids are eluted in decreasing order of their mol. wt., and, under standardised conditions, each acid can be identified by its rate of elution.

Each fraction of eluate is concentrated to a vol. of 2 to 3 ml by distillation of the solvent and titrated under N with 0·005 *N* Na ethoxide. The scale of operation requires about 0·2 to 2·0 ml of 0·005 *N* NaOH for each constituent, and the column contains 0·4 g of silica gel. W. C. JOHNSON

1874. **Chromatographic separation of higher fatty acids on cellulose acetate paper.** F. Micheel and H. Schweppe (*Angew. Chem.*, 1954, **66** [5], 136–137).—Saturated straight-chain (C_5 to C_{18}) fatty acids can be separated on cellulose acetate paper as the K salts of the corresponding hydroxamic acids in ethyl acetate - tetrahydrofuran - H_2O (0·6:3·5:4·7 by vol.) and identified by the intensive coloration with $FeCl_3$. R. C. MURRAY

1875. **Some applications of glycolic titration. II. Estimation of salts of organic acids.** M. N. Das (*J. Indian Chem. Soc.*, 1954, **31** [1], 39–42).—Acetates of Ca, Ba, Zn, Mn, Pb and Hg have been titrated with acid in ethylene glycol - *iso*propanol solvent (1:1), both potentiometrically and with an indicator, the percentage error being $\gtrless$ 0·7. HCl or $HClO_4$ are equally satisfactory, except for Hg for which the latter is unsuitable. In presence of excess of NaCl, $HClO_4$ can be used and the possibility of estimating inorganic acid halides by titration with $HClO_4$ in a glycolic solvent in presence of excess of mercuric acetate is thus indicated. M. TADMAN

1876. **Peak volume - concentration relationships with progressively changing solvents in organic acid chromatography.** C. Mader (*Anal. Chem.*, 1954, **26** [3], 566–567).—The organic acids were separated by a silicic acid column in a *n*-butanol - $CHCl_3$ mixture as mobile phase, the solvent mixture being progressively changed. The peak volume - concn. relationships were calculated for the individual acids, and the results showed that the peak volume concn. for each acid can be expressed by a general equation, the characteristics of which, for each particular acid, are useful for its identification. The acids studied were aconitic, oxalic, malic, citric and tartaric. G. P. COOK

1877. **Minimum error titrations of mixtures of two weak acids.** A. Frisque and V. W. Meloche (*Anal. Chem.*, 1954, **26** [3], 468–471).—Binary acid mixtures that give titration curves with a single sharp inflection can often be analysed by use of the individual pK values in the calculations. A mathematical treatment is presented for the location of a pH value, which represents a point of min. error, to be used in calculating the concn. of 2 weak acids giving one break on titration. Under the conditions described, it is only necessary to determine the volume of the titrant at the point of min. error and at the point of complete neutralisation. Experimental verification is presented with a mixture of benzoic and *m*-nitrobenzoic acids. G. P. COOK

1878. **Potentiometric non-aqueous titration of substituted fatty acids.** J. Radell and E. T. Donahue (*Anal. Chem.*, 1954, **26** [3], 590–591).—The acid sample or benzoic acid standard, containing acid approx. equivalent to 7 ml of 0·1 *N* Na methoxide, is dissolved in 50 ml of benzene - methanol (3 + 1) solvent and 0·2 g of LiCl is added. The soln. is titrated potentiometrically at calomel and antimony electrodes. The neutralisation equivalents for nine fatty acids are listed. The method has been used to analyse wool-wax fatty acids. G. P. COOK

1879. **Test for acyclic carboxylic acid anhydrides.** D. Davidson (*Anal. Chem.*, 1954, **26** [3], 576).—To 1 ml of a 3 per cent. soln. of 1-amino-1-*p*-nitrobenzoylphenylacetic acid in pyridine, add 1 drop of the substance to be tested. Acyclic carboxylic anhydrides develop a blue colour within a few seconds. Acids as strong as salicylic acid inhibit the colour formation, but inhibition can be overcome by adding a few drops of triethylamine. The results are positive with many other acid anhydrides. G. P. COOK

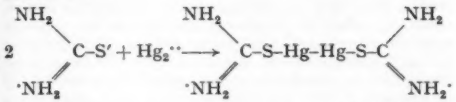
1880. **Preparation and estimation of dimers of ketens of normal long-chain fatty acids.** S. Piekarski (*Compt. Rend.*, 1954, **238**, 1241–1243).—Diketens of *n*-octoic, lauric, myristic and palmitic acids are prepared from triethylamine and the corresponding acid chloride ($R{\cdot}CH_2{\cdot}COCl$). The reactions of these diketens with primary amines at room temp. to form β-ketonic amides are used for the gravimetric estimation of diketens. Dissolve the sample (0·4 to 1 g) in anhyd. ether or hexane (20 to 35 ml), add an excess of ethylamine, distil off the solvent and excess of ethylamine and weigh the ppt. (dried for 45 min. at 110° C) to within 0·02 mg. The diketen content is calculated from the wt. of amine fixed and the mol. wt. of the fatty acid. W. J. BAKER

1881. **Electrometric titration of small quantities of oxalic acid.** S. Samson with H. Zschuppe (*Chem. Weekbl.*, 1954, **50** [19], 341–345).—Small quantities of oxalic acid in vegetable materials are determined by titration with a 3·5 per cent. soln. of hexanitratoammonium cerate in 1·4 *N* nitric acid to a dead-stop end-point. The reagent soln. is prepared by heating cerium oxalate until chocolate brown, dissolving it in conc. nitric acid, diluting with an equal volume of water and adding an excess of NH_4NO_3. The soln. is evaporated until crystals appear and then cooled. After drying, 3·5 g of the crystals are dissolved in 1 litre of 1·4 *N* HNO_3, giving a 0·0037 *N* soln. This soln. is stable for some days. A potential of 200 mV is used. When more dilute solutions are used, the result of a blank determination should be used as a correction. Quantities of oxalic acid of the order 0·0025 mg equivalents can be determined satisfactorily. G. MIDDLETON

1882. **Determination of vinylidene cyanide.** W. P. Tyler, D. W. Beesing and S. J. Averill (*Anal. Chem.*, 1954, **26** [4], 674–677).—The method is based on the reaction of vinylidene cyanide with anthracene in anhydrous toluene solution and measurement of the excess of anthracene at 359 mμ by differential colorimetry. The standard deviation is $\pm$ 4·1 parts per 1000 for all ranges of purity; accuracy is good. Most polar compounds, especially water and organic bases, must be absent, as these polymerise the cyanide before reaction with the anthracene can occur. Some kinetic data for the reaction in benzene soln. are also given and the equilibrium constant of formation of an anthracene - vinylidene cyanide complex in non-polar solvents has been estimated for one set of conditions. G. P. COOK

1883. **Polarography of thiourea.** R. L. Edsberg (*Anal. Chem.*, 1954, **26** [4], 724–726).—Polarograms of thiourea at one concn. run at pH 4 and pH 6 have the same half-wave potentials. There is therefore no reaction involving H^+. The half-wave potentials of polarograms run on various concn. of thiourea shift to more negative values with increasing concn. It is therefore probable that a complex or insol.

reaction product with Hg is formed. The probable reaction is—



A. J. MEE

1884. **Molar absorptivity of dithizone in chloroform.** A. S. Landry and S. F. Redondo (*Anal. Chem.*, 1954, **26** [4], 732–733).—Solutions of pure dithizone of known concn. can be prepared by stripping a dithizone - $CHCl_3$ soln. twice with dil. aq. NH_3 by using $\simeq$ 10 times as much aq. NH_3 as dithizone soln. in each extraction. After removal of the $CHCl_3$ layer, the filtered aq. phase is neutralised with dil. pure HCl (1 + 1), and the pptd. dithizone is then re-extracted into pure $CHCl_3$. The mol. concn. is calculated as: observed absorbancy at 606 mμ/mol. absorptivity at 606 mμ. The mol. absorptivity of dithizone in $CHCl_3$ (as determined from 25 replicate observations and involving formation of non-absorptive Hg^{II} - dithizone complex) is $(40{\cdot}6 \pm 0{\cdot}5) \times 10^3$. Procedure for preparing standard solutions containing 4·4 and 7·1 mg of dithizone per litre, respectively (for use in determinations of Cd, Pb and Hg), is described. The method avoids the preparation of pure solid dithizone. W. J. BAKER

1885. **Partition chromatography of a homologous series of volatile primary amines.** R. A. Clayton and F. M. Strong (*Anal. Chem.*, 1954, **26** [3], 579–580).—The system described gives good resolution of a series of C_2 to C_8 primary amines. The column is packed with Celite 545 and the stationary phase consists of 18 ml of methanol, 3 ml of water and 2 ml of ethanolic phenolphthalein (which serves as a column indicator) diluted to 1 litre with water. The mobile phase is light petroleum equilibrated with the stationary phase in the preparation of the column. The identification of the amines in the eluate fractions is established by paper-chromatographic comparison with known samples in *n*-butanol - acetic acid - water (48:2:50) as solvent. G. P. COOK

1886. **Carbon dioxide content of ethanolamine solutions.** P. T. Wagner and M. L. Lew (*Anal. Chem.*, 1954, **26** [3], 575–576).—Add 35 ml of 0·5 *N* NaOH (CO_3'' free) to an Erlenmeyer flask containing $\simeq$ 5 g of Na acetate and 5 ml of the ethanolamine sample. Heat to boiling, cool and add 120 ml of 95 per cent. ethanol plus 1 ml of alizarin yellow indicator. Titrate to a light orange end-point using 0·5 *N* acetic acid in 95 per cent. ethanol, matching the colour against that obtained by titrating the NaOH and the reagents with the acetic acid. The CO_3'' content is calculated from the back-titration titre. The error is ± 0·04 g of CO_2 per 100 ml of sample. G. P. COOK

1887. **The determination of phenols with 1-fluoro-2:4-dinitrobenzene. II. Splitting of dinitrophenyl ethers by amines.** H. Zahn and R. Kockläuner (*Z. anal. Chem.*, 1954, **141** [3], 183–188).—A previous publication (Zahn and Würz, *Brit. Abstr. C*, 1952, 202) describes the preparation of dinitrophenyl ethers from phenols by the action of 1-fluoro-2:4-dinitrobenzene. This paper describes a method of recovering the phenol in about 80 per cent. yield by heating the ethers with an amine; the amine becomes attached to the 2:4-dinitrobenzene ring. Hexamethylenediamine is particularly suitable, but amino-acids can also be used. Experimental details are given for the preparation of 2:4-dinitrophenylglycine, 2:4-dinitrophenylvaline, and ε-dinitrophenyloxylysine from an appropriate phenyl ether and amino-acid; the corresponding phenols are also formed. P. S. STROSS

1888. **Some applications of glycolic titration. I. Estimation of organic bases.** M. N. Das and S. R. Palit (*J. Indian Chem. Soc.*, 1954, **31** [1], 34–38).—Ethylene glycol - *iso*propanol (1:1) was successfully used as a solvent in the titration of some alkaloids, amino-acids and aminophenols. The titration curves show the base strength of substituted anilines to be as follows: *p*-aminophenol > *o*-aminophenol > *p*-aminosalicylic acid > aniline > anthranilic acid. M. TADMAN

1889. **A new colorimetric method for the micro-determination of aniline and its compounds.** K. W. Merz and A. Kammer (*Arch. Pharm., Berlin*, 1953, **286** [4], 198–205).—The method was devised to determine *N*-phenyl*iso*propylurethane (after hydrolysis) in potatoes. It is based on the reaction of oxidised *p*-phenylenediamine with aniline to give coloured indamines, which are extracted with CCl_4. Coloured compounds produced in the absence of aniline are not extracted in this way. To 20 ml of the aniline-containing solution at pH 5 to 6 (produced, say, by hydrolysis at 145° to 150° C of *N*-phenyl*iso*propylurethane with conc. HCl), add 2 ml of 0·2 per cent. *p*-phenylenediamine hydrochloride followed by a cold 2·5 per cent. soln. of potassium persulphate and shake; after exactly 10 min., add to the blue solution 10 ml of CCl_4 and 3 ml of 2*N* NaOH; separate and filter the red CCl_4 layer after 2 min. shaking, and measure the extinction at $\simeq$ 500 mμ. Calculate the aniline content from a calibration curve. From 20 to 1600 μg can be determined with an accuracy of ± 3 per cent. Alcohols interfere and must be absent. P. S. STROSS

1890. **Iodimetric determination of phenylhydrazine.** P. Endroi (*Magyar Kém. Folyóirat*, 1953, **59**, 211–212).—To the sample to be examined, add 20 ml of distilled water, previously boiled and cooled, 2 ml of 10 per cent. HCl in a 100-ml measuring flask and water to make 100 ml. To 10 ml of this soln., add 20 ml of saturated aq. Na_2HPO_4 soln. and titrate with 0·1 *N* I soln., add as much I soln. as was used for the titration, add 20 ml of saturated aq. Na_2HPO_4 soln., set aside for 5 min., acidify with 4 ml of 10 per cent. HCl, add 1 ml of 1 per cent. starch soln. and titrate with 0·1 *N* $Na_2S_2O_3$. CHEM. ABSTR.

1891. **Paper chromatography of some substituted napthaquinones.** T. Sproston, jun., and E. G. Bassett (*Anal. Chem.*, 1954, **26** [3], 552–553).—An exhaustive study was made of solvents and chromogenic sprays to establish accurate R_F and colour values for many different 1:4-naphthaquinones and benzoquinones. The most satisfactory solvent mixture was composed of pentanol, pyridine and water (6:4:3 by vol.) and the spots were developed with 5 per cent. aq. NaOH. R_F values were markedly influenced by the presence of ·OH, CH_3·, CH_3O· and NH_2· radicles. Colours and R_F values for 37 compounds are listed. G. P. COOK

1892. **Spectrophotometric determination of the purity of anthracene.** D. Ciecierska, K. Gorczynska and J. Swietoslawska (*Przem. Chem.*, 1953, **32** [12],

616–623).—Ultra-violet absorption curves were established for pure anthracene, carbazole and phenanthrene and also for mixtures of these compounds. These curves were used to determine the purity of various grades of technical and purified anthracene with the aid of a Beckman model DU spectrophotometer, according to the Beer - Lambert law. The products examined contained 85 to 100 per cent. of anthracene, the impurities consisting of carbazole, phenanthrene and small quantities of other substances. The relative exactness of the determination of anthracene is 0·3 to 0·8 per cent. An amount of up to 10 per cent. of carbazole in technical anthracene can be estimated with a relative exactness of 4 to 9 per cent., and the sum of phenanthrene and carbazole in mixtures with anthracene is established with an accuracy of 10 per cent. The max. relative error in the separate estimations of carbazole and phenanthrene is 10 per cent.

H. BURSTIN

1893. **Chromatographic separation of acid obtained from oxidation of α-pinene.** D. E. Baldwin, V. M. Loeblich and R. V. Lawrence (*Anal. Chem.*, 1954, **26** [4], 760–762).—The ten or eleven mono- and dicarboxylic acids in the crude oxidation product of α-pinene can be reasonably well separated and identified by partition chromatography (*i*) by a modified Marvel - Rands method (*J. Amer. Chem. Soc.*, 1950, **72**, 2642) in which the polarity of the mobile phase ($CHCl_3$) is increased in increments of 1 per cent. of *n*-butanol or (*ii*) by a method involving H_2O on silicic acid as immobile phase with benzene and benzene - *n*-butanol as mobile phase. The samples are dissolved in water-saturated $CHCl_3$ or benzene, and the silicic acid columns (60 cm by 18 mm) are carefully standardised (against propionic acid) to give the required "peak effluent vol." for each acid (values listed). The percentages of the individual acids present in the sample are calculated from the amount of acid in each fraction forming the peak (determined by titration with 0·01 *N* NaOH). In absence of pinononic acid, pinonic and norpinic acids are almost completely separated by method (*i*), which also effects complete separation of the dicarboxylic pinic and norpinic acids. The mono- and dicarboxylic acids together are effectively separated by method (*ii*), which, besides separating terpenylic and homoterpenylic acids, can also be used for samples of crude pinic acid. Chromatograms of acid mixtures treated by the above methods are shown.

W. J. BAKER

1894. **Colorimetric determination of pyridine-2:5-dicarboxylic acid.** Kuang Lu Cheng and J. A. Riddick (*Anal. Chem.*, 1954, **26** [3], 536–538).—Pyridine-2:5-dicarboxylic acid (**I**) is determined by measurement, at 415 mμ, of the coloured complex formed with Fe^{++}. The optimum pH for the reaction is 3·5 to 4·0 and the colour is stable for $\simeq$ 6 hr. The accuracy is $\gtrless \pm$ 1·8 per cent. and the precision is 0·2 per cent. or better, Beer's law being obeyed over the range 0 to 12·5 mg per 25 ml of soln. The method is applicable to the determination of **I** in the presence of mixtures of HNO_3, 5-ethyl-2-methylpyridine and pyridine-3-carboxylic acid.

G. P. COOK

1895. **Rapid determination of tetra-ethyl lead in motor spirit.** J. Smelik (*Prakt. Chem.*, 1954, **5** [1], 9–12).—Published methods are reviewed. After bromination and solution in aq. HNO_3, the Pb can be determined by anodic deposition by applying the empirical factor 0·860 for $Pb:PbO_2$. Slight incompleteness of the deposition may be compensated for by adding a known amount of Pb^{++} before electrolysis. For routine control, the soln. is compared with a standard by placing the two solutions in two cells connected in parallel to an adjustable p.d. *via* calomel electrodes and twin dropping electrodes. A null galvanometer between the two standard electrodes indicates any e.m.f. caused by a difference in concn. of Pb^{++} between the two solutions. This method of comparison can be applied directly to a solution of the spirit in 2-ethoxyethanol containing HCl (*cf. Brit. Abstr. C*, 1951, 91 and 192). The method is rapid (12 min.) and accurate for normal concn. of tetra-ethyllead in spirits that are not rich in unsaturated hydrocarbons.

A. R. PEARSON

1896. **Evaluation of twenty qualitative tests for petroleum sulphur compounds.** C. Karr, jun. (*Anal. Chem.*, 1954, **26** [3], 528–536).—An investigation of 20 different reactions on 15 different S compounds was made in order to determine their value as qual. tests. In the choosing or developing of these tests, reactions were sought that gave rapid coloration or pptn. with S concn. as low as 0·2 per cent. and did not involve separations and unusual reagents. Test procedures for aliphatic and aromatic thiols and disulphides, aliphatic, cyclic and aromatic sulphides, thiophens and condensed thiophens, are given. Some of these tests have been recommended by other workers, but approximately half are believed to be new.

G. P. COOK

1897. **Interferometric determination of benzene in coke oven gas.** W. Ohme (*Erdöl u. Kohle*, 1954, **7** [2], 73–77).—The instrument is calibrated and the refractivity of the benzene vapour is determined by comparing, against N as a standard, gases of known refractive index and N carrying known concn. of mixed hydrocarbons from the rich and stripped gas streams; the respective mol. wt. of these hydrocarbons are 84·3 and 92. It is assumed the refractive index is additive. The coke oven gas to be tested is compared against N before and after the removal from it by active C of all hydrocarbons containing $\not< 5$ C atoms. As each hydrocarbon saturates and breaks through the active C, it makes a contribution $(n' - 1)x$ to the total value of n, where n' is its refractive index and x its concn. in vol. per cent. As x is affected by the displacement of the portion previously adsorbed, the original concn. of each hydrocarbon cannot be calculated from the position of the step in n. The total $\not< 5$ C hydrocarbons can however be calculated as vol. per cent. and converted to g per cu. metre if the mean mol. wt. (M) is found, *e.g.*, from the f.p. lowering of *p*-dichlorobenzene by a sample of the mixed hydrocarbons recovered by active C. The value of M for rich and stripped gases showed little day-to-day change.

A. R. PEARSON

1898. **A note on the modified Gray - King assay of oil shale.** G. E. Mapstone (*J. Inst. Petrol.*, 1953, **39**, 848–50).—The effect of using solid CO_2 for cooling the oil receiver in the Gray - King assay is compared with that obtained with receivers maintained at 0 to 1° C and at 22° C (mean summer temp.). Figures are given for seven assays on shales varying in content from 33·7 to 183·7 gallons per ton. The differences between cooling at 22° C and in an ice-bath are slight, but results are considerably lower than the assay with solid CO_2. These losses of lower boiling naphtha become greater as yields increased. The figures given enable corrections to be made for converting yields at the higher temperatures.

D. JENKINS

1899. **Rapid method and apparatus for analysis of technical emulsions.** H. Kohnle (*Bitumen, Teere, Asphalte, Peche*, 1954, **5** [3], 72–73).—Water, fillers and disperse material (bitumen, synthetic resin, etc.) are determined by weighing a sample of the emulsion into an extraction thimble that is placed in the neck of a flask containing a suitable solvent (such as toluene) and connected to a Dean - Stark head. If the sample leaks through the thimble, an additional wrapping of filter-paper is applied or the solution finally obtained is centrifuged before recovering from it the bitumen or other sample material. A. R. PEARSON

1900. **Estimation of the moisture content of plant fibres.** M. A. Timonin (*Textile Ind., U.S.S.R.*, 1953 **13** [12], 13).—The dependence of the moisture content of fibres on the R.H. of the atm., temp. and pressure, and its industrial importance are discussed. A new formula, more accurate than the Müller formula, based on experimental work for cotton, flax and hemp is—

$$\frac{\phi}{a + \frac{bt}{100 - t}} \left[1 + \frac{(c - \phi)^2}{d}\right],$$

where ϕ is the R.H. of the atmosphere, t the temp. of the air, and a, b, c and d are constants having the respective values of 7·45, 2·0, 70 and 7000 for cotton, 6·43, 1·75, 65 and 4000 for flax and 6·0, 1·5, 65 and 4500 for hemp. Tables are given to show the close agreement between the moisture contents of the fibres as calculated by the new formula and as obtained experimentally over a range of R.H. values. The use of the formula to fix the proper R.H. for storage of raw flax and hemp and for storage of raw materials in textile factories is demonstrated with the aid of a graph.
H. L. WHITEHEAD

1901. **Identification of textile and related fibres.** J. E. Ford and W. J. Roff (*Shirley Inst. Mem.*, 1954, **27** [6], 77–108).—A system for the identification of the commoner fibres is proposed. The sample is examined microscopically for homogeneity and if necessary, separated into components by hand sorting, solvent extraction, etc. Natural and man-made fibres are distinguished microscopically. Both types are further sub-divided into six groups, the former largely on morphological evidence and the latter on thermoplasticity and the presence of N or Cl. Confirmatory tests are given for individual fibres within these groups. The effect of manufacturing processes is taken into account. Some 40 natural and 30 man-made fibres are considered, and 64 photo-micrographs are included. A. M. SPRATT

1902. **Synthetic fibres. Properties and microscopic identifying reactions.** W. Bobeth (*Faserforsch. Textiltech.*, 1954, **5** [3], 115–129).—A synopsis is given of the most important synthetic fibres under the headings of polyvinyl chloride, mixed polymer, polyacrylonitrile, polyamides, polyurethanes and polyesters. Their physical properties (sp. gr., dry tenacity, relative wet strength, stretch, moisture absorption, m.p., softening point and angle of transverse brittleness), aspect under the microscope (longitudinally and in section), behaviour to solvents and swelling agents, and industrial uses are given. The characteristic microscopic behaviour of the different fibres towards test solvents and reagents [acetone retarded with ethanol (5 + 1), toluene retarded with ethanol (1 + 1), $CHCl_3$, CH_2Cl_2, dimethylformamide, $CHBr_3$ or dioxan retarded with ethanol, phenol soln. retarded with nitrobenzene, $ZnCl_2$ - I soln. retarded with H_2O, conc. HNO_3 retarded with H_2O] is given and discussed with photo-micrographs of the treated fibres and of their cross-sections. H. L. WHITEHEAD

1903. **Fluidity measurements on cellulose solutions by the nitrate method.** F. H. Robaard (*Chem. Weekbl.*, 1954, **50** [17], 297–307).—The literature on the relationship between the degree of polymerisation of cellulose and the fluidity of solutions is reviewed, the nitrate method, in which the cellulose is first nitrated, being compared with the direct cuoxam and cupri-ethylenediamine methods. Results are given of experiments carried out to determine the effect of variations in the composition of the nitrating acid, time of nitration and moisture content of sample on values obtained by the nitrate method. Butyl acetate is used as a solvent for the nitrated cellulose (rayon 250 mg per 100 ml, cotton 50 mg per 100 ml); the solutions remain stable for over 20 days. Graphs and tables are given for converting fluidities and intrinsic viscosities obtained by the nitrate method on rayon and cotton to the corresponding cuoxam values. E. HAYES

1904. **Solubility in acid as a means of determining changes in wool.** H. Zahn and A. Würz (*J. Text. Inst.*, 1954, **45** [3], P88–P92).—An acid-solubility test, analogous to the alkali-solubility tests of Harris and Smith, is proposed as a test for changes in the structure of wool. Untreated wool subjected to 4 *N* HCl under the conditions of the Harris - Smith test—1 hr. at 65° C (liquor: wool ratio, 100: 1)—suffers a loss in weight of about 12 per cent. It is shown that treatments of wool can be classified according to their effects on the acid- and alkali-solubilities of the wool, *e.g.*, (*i*) cross-linking reduces both acid- and alkali-solubility, (*ii*) chlorination, treatment with alcoholic KOH, etc., increases both acid- and alkali-solubility, (*iii*) heating to 100° C increases acid- and reduces alkali-solubility and (*iv*) treatment with dinitrophenol decreases acid-solubility and increases alkali-solubility.
L. VALENTINE

1905. **Determination of fatty oils in citronella oil.** A. I. Biggs (*Anal. Chem.*, 1954, **26** [3], 602–603).—The method is based on the saponification of any fatty oils in citronella oil and determination of the glycerol by oxidation to formaldehyde with periodic acid. The citronella oil is saponified with 0·5 *N* methanolic KOH for 1 hr., the residual soln. is diluted with saturated NaCl (made slightly acidic with H_2SO_4) and the methanol is evaporated off. The aq. soln. is extracted with ether, made alkaline, extracted again with ether, made neutral and extracted twice more with ether. The aq. soln. is warmed and diluted with water to give a concn. of 0·001 to 0·006 per cent. of glycerol. Two ml of this soln. are added to 0·2 ml of KIO_4 (1·25 per cent.) in *N* H_2SO_4 and shaken for 10 min. Freshly prepared 1 per cent. aq. phenylhydrazine hydrochloride (0·5 ml) is added, and the mixture is shaken and set aside for 10 min. After addition of 0·5 ml of 1 per cent. $FeNH_4(SO_4)_2$, the mixture is shaken and 25 ml of 6 *N* H_2SO_4 are added quickly, the mixture is well shaken and the optical density is measured at 520 mμ. The colour is stable for 15 min. The error is < 5 per cent. G. P. COOK

1906. **An apparatus for detecting plasticiser in polymethyl methacrylate sheet.** J. Haslam and S. Grossman (*Analyst*, 1954, **79**, 238–242).—The apparatus described and illustrated provides a rapid method for discovering whether or not polymethyl

methacrylate sheet is plasticised without subjecting the sheet to damage. Light from a mercury-vapour lamp is passed through a condensing lens and filter and thence into a simple u.v. spectroscope where it illuminates a fluorescent screen. Test sheets of the plastic placed in the light path show differences in light absorption according to whether they are plasticised or not, and these differences are readily seen with the spectroscope. The apparatus serves also for the detection of u.v. light absorbers.

A. O. JONES

1907. **Identification of polyhydric alcohols in polymeric esters.** J. F. Shay, S. Skilling and R. W. Stafford (*Anal. Chem.*, 1954, **26** [4], 652–656).—A 10-g portion of the polyester is dissolved in benzene or acetone and saponified with 250 ml of $\simeq$ 0·5 *N* alcoholic KOH. The insol. K salts are filtered, the filtrate is made slightly acid with HCl and the pptd. KCl is removed by filtration. The filtrate is concentrated to 5 ml, made up to $\simeq$ 20 ml with water and is extracted with an equal vol. of ether. The aq. phase is made slightly alkaline and is evaporated to dryness. The residue is extracted with ethanol, filtered and the filtrate is evaporated in a stream of dry air. This residue represents the polyhydric alcohol fraction. The alcohols are identified by i.r. analysis by using the spectra of commercial alcohols as standards. The method is effective for individual glycols and for mixtures of 2 liquid glycols. Mixtures of liquid and solid glycols require further fractionation before identification. Spectra of 28 polyalcohols are illustrated.

G. P. COOK

1908. **Identification of dicarboxylic acids in polymeric esters. Preparation and properties of diethyl esters and potassium salts.** R. W. Stafford, J. F. Shay and R. J. Francel (*Anal. Chem.*, 1954, **26** [4], 656–661).—The diethyl esters of adipic, azelaic, citraconic, fumaric, itaconic, maleic, phthalic and sebacic acids were prepared and purified and the following physical properties were determined on the purified products: (*i*) vapour pressure at 50 mm of mercury, (*ii*) saponification values, (*iii*) u.v. absorption spectra, (*iv*) i.r. spectra, (*v*) refractive indices and (*vi*) densities. The dipotassium salts were prepared from the esters, and the Kappelmeier procedure was evaluated in the process. Both the diethyl esters and the dipotassium salts were found to be characteristic derivatives for the identification of dicarboxylic acids, the i.r. and u.v. spectra giving the most determinative properties. The esters permit more effective resolution than the salts when mixtures of acids were involved, and the Kappelmeier procedure for phthalic acid was not generally applicable as a quant. method.

G. P. COOK

1909. **Tracer method for sulphur solubility and diffusivity in rubber.** I. Auerbach and S. D. Gehman (*Anal. Chem.*, 1954, **26** [4], 685–690).—S tagged with ^{35}S is brought into intimate contact with rubber, the S diffusing into the rubber until saturating it. The activity of the rubber on the opposite side is measured and is converted to S concn. by reference to a calibration graph. The effect of a number of compounding ingredients, including carbon black, on apparent S solubility is also given. The solubilities in 26 rubbers (base and compounded) are listed. The procedures described are applicable to determining the diffusivity of S and ^{35}S or ^{14}C-tagged compounds in rubber; the blooming characteristics of various rubbers and mixtures can also be determined by similar means. G. P. COOK

1910. **Measuring wet gel strength of coagulant-dipped films. Paper dip method.** A. I. Medalia, H. B. Townsend and H. N. Grover (*Anal. Chem.*, 1954, **26** [4], 697–703).—The method is based upon the use of absorbent paper of low wet strength, impregnated with $Ca(NO_3)_2$, as a base for the deposition of a latex film. The paper is dipped for 30 sec. and then removed from the latex together with the adhering coagulant film. This film is rinsed in water and then mounted on a special tensile testing apparatus designed for high elongation and low tensile strength. Elongation and tensile studies on natural and synthetic latexes and the effects of pre-vulcanisation, compounding and dilution of these latexes are given. Tensile properties of film from a given latex have been measured as a function of time and of conditions of drying after deposition. Properties of 15 samples of raw latex films are listed. G. P. COOK

See also Abstracts 1762, 1826, 2004, 2006, 2014, 2016.

4—BIOCHEMISTRY
INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Blood, Bile, Urine, etc.

1911. **A simple indirect method for determining plasma sodium levels.** M. J. Krainin, V. S. Whitner and A. J. Merrill (*J. Lab. Clin. Med.*, 1943, **43** [3], 482–488).—The method is based on the relationship between erythrocyte volume and plasma sodium concentration. The initial haematocrit value is read on the sample of heparinised blood; a known hypertonic and a known hypotonic solution are then substituted for part of the plasma in each of replicate determinations and the new haematocrit values are determined. A value calculated from the observations is plotted against plasma sodium concentration and gives a rectilinear calibration, the co-ordinates for which are given. The method requires little apparatus and is sufficiently accurate for clinical use. W. H. C. SHAW

1912. **The quantitative flame-photometric semi-micro-determination of calcium in 0·1 ml of deproteinised whole blood.** G. O. Schlütz (*Schweiz. med. Wochschr.*, 1953, **83**, 452).—Mix 0·1 ml of finger-tip blood with 0·1 ml of 20 per cent trichloroacetic acid and 0·3 ml of H_2O. Spin in a centrifuge and wash the ppt. with 0·5 ml of H_2O. Combine the supernatant liquids and boil down to 0·3 ml to decompose the trichloracetic acid. Adjust to pH 5 with 0·1 per cent. acetic acid, add 0·1 ml of cold saturated ammonium oxalate and let stand for 30 min. Centrifuge and wash the ppt. twice with 2 per cent. ammonia soln. for removal of Na and K. Take up the ppt. in 0·5 ml of 0·1 *N* HCl and measure on a flame photometer against a standard Ca soln. prepared from vacuum-dried $CaCO_3$.

CHEM. ABSTR.

1913. **Complexone titration of calcium in serum.** K.-H. Kimbel (*Hoppe-Seyl. Z.*, 1953, **293** [6], 273–277).—A satisfactory technique is described for the use of complexone in the titration of Ca^{++} in clinical samples, using Eriochrome black T as indicator. The reaction is carried out at pH 11·5.

P. G. MARSHALL

1914. **A method for the volumetric determination of iron in blood with the aid of complexone III.** E. Häberli (*Experientia*, 1954, **10** [1], 34–35).—Heat

1·00 ml of blood in a micro-Kjeldahl flask with conc. H_2SO_4 (2 ml), conc. HNO_3 (2 ml) and conc. $HClO_4$ (5 drops). As soon as the water is evaporated and the soln. begins to darken, add HNO_3 (15 drops) and $HClO_4$ (2 drops) and heat until the nitrogen oxides and water are removed. Heat more strongly until the soln. is bright yellow and clear and the H_2SO_4 is almost completely removed. Cool, add 2 *N* NaOH (10 drops), and neutralise further with conc. NaOH soln. until on shaking the pptd. $Fe(OH)_3$ just redissolves,; then make slightly alkaline with dilute NaOH soln. Redissolve the ppt. in 2 *N* HCl. The vol. is now 2 to 3 ml. Warm to 50° C, add 30 to 50 mg of catechol-3:5-disulphonic acid ("Tiron") and titrate the deep blue-green soln. with 0·01 *M* complexone III soln. until yellow. In 16 tests with defibrinated dog blood, a mean value of 44·2 mg of Fe per 100 ml with a standard deviation of ± 0·93 mg per cent. was found. N. E.

1915. **Determination of blood sugar with anthrone.** S. J. Prokhovnik and J. F. Nelson (*Aust. J. Exp. Biol.*, 1953, **31** [3], 279–282).—The use of Dreywood's (*Brit. Abstr. C*, 1946, 280) anthrone reagent has certain advantages over the Hagedorn - Jensen method; it is more sensitive and specific for carbohydrates and gives a closer approximation to the true glucose concn. than methods measuring all reducing substances. Interference from non-carbohydrate sources is negligible. The method is suitable for 6 to 100 μg of glucose, the precision being greatest for 40 to 60 μg. It is not affected by the anti-coagulants commonly used for blood preservation. N. E.

1916. **Improved micro-determination of reducing sugars with ferricyanide according to the method of Fujita Akiji and Danko Iwatake.** D. Wolszlegier (*Przem. Chem.*, 1953, **32** [12], 623–624).—The Fujita Akiji and Danko Iwatake modification of the Hagedorn - Jensen determination of reducing sugars has been rendered cheaper, without impairing its accuracy, by reducing the quantity of phosphate buffer soln. to 1/4 of that used previously and the quantity of the $ZnSO_4$ - KI soln. to 40 per cent. of that formerly used. In the improved procedure the solutions used are: (*i*) a buffer, 5·04 g of K_2HPO_4 and 15·3 g of K_3PO_4 in 1 litre of water, (*ii*) 20 g of $ZnSO_4$ and 100 g of NaCl in 1 litre of water and (*iii*) 4 g of KI in 1 litre of water. Three parts of (*ii*) are mixed with two parts of (*iii*) just before the test is carried out. H. Burstin

1917. **Modification of the Schönheimer and Sperry method for determination of cholesterol in serum.** E. Mertens and C. Albers (*Hoppe-Seyl. Z.*, 1953, **293** [6], 244–253).—Various methods of determination of cholesterol in serum are reviewed; that of Schönheimer and Sperry (with modifications) is recommended. Accurate determinations of free and total cholesterol are possible. P. G. Marshall

1918. **Chromatographic separation of the plasma lipids.** D. L. Fillerup and J. F. Mead (*Proc. Soc. Exp. Biol. Med.*, 1953, **83** [3], 574–577).—*Total plasma lipid*—The freshly taken blood was placed immediately into a 15-ml centrifuge tube containing a few specks of heparin-Na, shaken and centrifuged. To 2 ml of plasma, 30 ml of a (4 + 1) mixture of formaldehyde and methanol was added with vigorous stirring. After 30 min. the ppt. was centrifuged and the supernatant liquid was evaporated to dryness under N at reduced pressure at room temp. The residue was extracted with light petroleum, this extract was evaporated as before and weighed as total lipid. *Chromatography*—For about 50 mg of lipid, silicic acid was packed in a tube of 2-cm diam. to a height of 7 cm. The column was washed under reduced pressure with 3 column vol. (1 vol. ≃ 14 ml) of absolute methanol; then equal volumes of acetone, anhydrous peroxide-free ether and light petroleum (boiling range 60° to 70° C) were added. The total lipids were transferred to the column in light petroleum soln. and eluted in successive fractions as follows: (*i*) sterol ester—20 column vol. of 1 per cent. of ether in light petroleum; (*ii*) neutral fat—20 c. vol. of 4 per cent. of ether in light petroleum; (*iii*) sterol—15 c. vol. of 10 per cent. of ether in light petroleum; (*iv*) fatty acids—15 c. vol. of 50 per cent. of ether in light petroleum; and (*v*) phospholipid—15 c. vol. of 25 per cent. of methanol in ether. The amount of each fraction was determined by wt. Artificial mixtures gave recoveries of 96 to 100 per cent. One example of application is described in which elaeostearic acid was found exclusively in the neutral fat fraction after being fed as the methyl ester. H. F. W. Kirkpatrick

1919. **The estimation of creatinine in serum.** M. H. Roscoe (*J. Clin. Pathol.*, 1953, **6** [3], 201–207).—Various factors in the alkaline picrate method have been standardised and the following procedure has been adopted. Measure 3 ml of serum into each of 2 centrifuge tubes; to the first (*X*) add 3 ml of dist. H_2O and to the second (*S*) 3 ml of standard soln. [0·5 g of creatinine in 1 litre of 0·1 *N* HCl diluted (2 + 5) with dist. H_2O]. Mix and to each add 3 ml of 5 per cent. w/v sodium tungstate, mix and add 3 ml of 0·33 *N* H_2SO_4. After 10 min. centrifuge and filter the supernatant liquid. To 6 ml of each filtrate and to 6 ml of dist. H_2O (*B*) add 4 ml of a fresh mixture of equal parts of 0·75 *N* NaOH and saturated picric acid, mix and set aside for 25 to 30 min. Measure the absorption with No. 603 filters reading *X* against *B* and *S* against *X*. Creatinine, mg per 100 ml = (Reading of *X*/ Reading of *S* × 0·5). If there is insufficient serum for *S*, prepare a standard with 6 ml of the 0·5 g per litre creatinine soln. and 4 ml of alkaline picrate reagent and read against *B*. A correction is then made in the above calculation for lower readings given by creatinine in serum filtrates by multiplying by 1·11. If the concn. of creatinine is > 3 mg per cent., the serum filtrate must be diluted and a higher concn. of creatinine must be added to the serum for *S*; the filtrate is diluted similarly. H. F. W. Kirkpatrick

1920. **Studies on the validity of the Hooker - Forbes test for the determination of progesterone in untreated blood.** M. X. Zarrow and G. M. Neher (*J. Clin. Endocrinol.*, 1953, **13** [2], 203–209).—The possibility of oestrogens vitiating the assay of blood progesterone by the Hooker - Forbes method (*Endocrinology*, 1947, **41**, 158) is investigated. The effect of oestrogens in blocking the specific action of progesterone on the stromal nuclei of the mouse uterus is confirmed; it is shown that the effect depends upon the ratio of oestrogen to progresterone. In untreated blood the amount of oestrogen present is insufficient to interfere with the test, which is therefore assumed to be valid. No blocking action was found with pregnanediol and testosterone propionate. The sensitivity of the test enabled detection of 0·3 μg of progesterone per ml of soln. H. F. W. Kirkpatrick

1921. **Estimation of dinitro-o-cresol in blood.** E. F. Edson (*Lancet*, 1954, i [19], 981–982).—A

method that takes only a few minutes has been devised for emergency determination of dinitro-*o*-cresol (**I**) in the blood of workers exposed to poisoning. Add 1 ml of whole blood to 5 ml of ethyl methyl ketone, and then add about 1 g of a mixture of NaCl (9 parts) and anhyd. Na_2CO_3 (1 part) and shake vigorously for 30 sec. At levels of **I** above 10 μg per ml the solvent will be visibly yellow. Filter the extract and compare it in a standard comparator with discs containing from 5 to 45 μg of **I** per ml. Results by this procedure are within 4 to 5 μg per ml (usually lower) of those given by absorptiometric methods. Levels of 1 to 10 μg per ml are considered trivial, 10 to 20 appreciable, 20 to 30 unsafe, 30 to 40 likely to cause toxic effects, 40 to 50 dangerously high and over 50 critically dangerous. The method can also be used for butyldinitrophenol, a toxic chemical used for weed control. In plasma or serum the level is approx. double that in the corresponding whole blood.

H. F. W. KIRKPATRICK

1922. **The estimation of profibrinolysin.** R. R. Soicher, P. Richards and B. Rose (*J. Lab. Clin. Med.*, 1954, **43** [3], 495–500).—A method is described in which profibrinolysin is pptd. with half-saturated ammonium sulphate from dilutions of plasma in acetate buffer at pH 3·7. For dilutions between 1:250 and 1:1000, pptn. is quant. The ppt. is dissolved in borate buffer (pH 7·8) and activated with streptokinase, and the enzyme activity is determined by the lysis time of a standard fibrin clot under controlled conditions. An inverse log. relationship holds between lysis time and concn. of fibrinolysin over the range 0·5 to 3 Loomis units per ml (*Arch. Biochem.*, 1947, **12**, 1). The normal profibrinolysin level in human plasma is 400 to 900 Loomis units per ml. W. H. C. SHAW

1923. **Analysis of gases in blood with the mass spectrometer. III. A method for the determination of nitrous oxide in blood.** J. S. Hattox, J. M. Saari and A. Faulconer, jun. (*Anesthesiology*, 1953, **14**, 584–590).—Nitrous oxide is partially extracted from blood by equilibration with air containing 0·93 per cent. of argon as internal standard. The resultant mixture of gases is then analysed with the mass spectrometer and the N_2O in the blood is calculated. The error is $\pm$ 2 per cent.

CHEM. ABSTR.

1924. **Analysis of gases in blood with the mass spectrometer. IV. Total extraction of gases for determination of carbon dioxide and oxygen in blood.** R. T. Patrick, J. M. Saari, S. Possati and A. Faulconer, jun. (*Anesthesiology*, 1954, **15**, 95–102).—All gases are extracted from the blood sample with release of chemically bound O and CO_2, followed by vacuum extraction and drying of all dissolved gases. Argon is added as internal standard, the gas mixture is analysed and the original O and CO_2 are determined. The method is adaptable to samples containing ether and N_2O. Comparison of this method with van Slyke's method showed a root mean square difference of $\pm$ 3·5 per cent. for O and $\pm$ 1·95 per cent. for CO_2 determinations.

CHEM. ABSTR.

1925. **Determination of inulin in plasma and urine by the use of anthrone.** R. P. White and F. E. Samson, jun. (*J. Lab. Clin. Med.*, 1954, **43** [3], 475–478).—A new rapid photometric method based upon colour reaction with anthrone is described. Glucose, which interferes, is removed by preliminary yeast treatment.

Procedure—Prepare a 20 per cent. aq. suspension of thoroughly washed bakers' starch-free yeast. This may be kept in a refrigerator for several days, but the yeast must be washed again on each day of use and the suspension must be remade. To 1·00 ml of oxalated plasma in centrifuge tube, add 3 ml of yeast suspension. Incubate at room temp. for 15 to 20 min., shaking occasionally. Centrifuge, decant the supernatant portion into a second tube and add 1 ml of 25 per cent. w/v aq. trichloroacetic acid to the supernatant liquid. Mix and set aside for 1 min. Wash the inside of the tube with 0·5 ml of water and then centrifuge. Decant the supernatant liquid into a test tube marked at 6·0 ml, and dilute to the mark. Transfer 4 ml to a 25 × 200-mm Pyrex-glass tube, add 8 ml of anthrone reagent (200 mg in 100 ml of conc. H_2SO_4), swirling vigorously. Cool in air to room temp. and measure the optical density at 620 mμ. Urine samples are diluted until the expected level is about that of plasma and are then assayed similarly. The calibration graph is prepared from known quantities of inulin added to control plasma or diluted (about 1 + 50) control urine; it is linear up to 0·8 mg per ml of sample. W. H. C. SHAW

1926. **Simplified procedure for separating porphyrins from urine by paper chromatography.** L. M. Corwin and J. M. Orten (*Anal. Chem.*, 1954, **26** [3], 608–609).—The total porphyrins are adsorbed from the urine on a Pb ppt. and then eluted with 12 to 15 per cent. HCl. An aliquot of the eluate is evaporated in a stream of air and the residue is taken up in 10 *N* aq. NH_3 (0·2 ml). This soln. is then spotted on a filter-paper strip and chromatographed in a developing solution of a 2:4-lutidine or 2:6-lutidine - water (1 + 1). The red fluorescent zones of porphyrins are observed under u.v. light.

G. P. COOK

1927. **The filter-paper electrophoretic identification of urinary chorionic gonadotrophin. A new test for pregnancy.** H. M. Stran and G. E. S. Jones (*Bull. Johns Hopkins Hosp.*, 1953, **93**, 51–53).—Chorionic gonadotrophin is excreted in early pregnancy, but is not found in the urine of non-pregnant women. The protein fractions of the urine from women in the first trimester were suspended and added to Whatman 3-mm filter-paper strips. These were studied electrophoretically (20 V per cm, pH 8·6, molarity 0·005, 20° C) during a 2-hr. migration. Nineteen of 20 samples contained a protein that migrated to the cathode. Samples of urine from non-pregnant women failed to show this protein. Such a filter-paper electrophoretic method might be adapted as a clinical test for pregnancy. CHEM. ABSTR.

1928. **The determination of pyramin.** W. O. Caster, O. Mickelsen and A. Keys (*J. Lab. Clin. Med.*, 1954, **43** [3], 469–474).—Pyramin, a pyrimidine metabolite of thiamine excreted in urine, is determined by a yeast fermentation method after destruction of thiamine by sulphite treatment. The pyramin values are calculated from the CO_2 evolved under standard conditions of fermentation, which are described in detail. The values are expressed in terms of 4-amino-5-ethoxymethyl-2-methylpyrimidine hydrochloride, which is used as the standard. The method is particularly useful for studying low thiamine intake levels, but gives abnormal values if heat degradation products of thiamine are present in the diet. The relation between thiamine intake and pyramin excretion level is discussed. W. H. C. SHAW

1929. **Spectrophotometric method for determination of urea.** G. W. Watt and J. D. Chrisp (*Anal. Chem.*, 1954, **26** [3], 452–453).—Urea is determined by the yellow-green colour produced when it is treated with *p*-dimethylaminobenzaldehyde (**I**). Ten ml of the reagent (2 g of **I**, 100 ml of 95 per cent. ethanol and 10 ml of conc. HCl) are added to appropriate urea aliquots and the vol. is made up to 25 ml with water. The absorption is measured at 420 mμ, Beer's law applying at concn. up to 320 p.p.m. The relative error is 1 per cent. over the range of 50 to 240 p.p.m. N_2H_4 and semicarbazide interference can be suppressed. G. P. Cook

1930. **Micro-determination of chemical elements in biological and other fluids by high-frequency discharge photometry.** G. O. Schlütz (*Hoppe-Seyl. Z.*, 1953, **293** [6], 254–256).—The construction of a new high-frequency discharge burner is described and illustrated. This renders possible continuous blood investigations of the smallest laboratory animals in large-scale experiments, without undue loss of blood. P. G. Marshall

1931. **Determination of arsenic in biological materials.** R. J. Evans and S. L. Bandemer (*Anal. Chem.*, 1954, **26** [3], 595–598).—The biological material is mixed with saturated $Mg(NO_3)_2$ soln. in a crucible lined with filter-paper, ignited over a burner or evaporated to dryness (depending on the sample) and then heated overnight at 600° C. The ash is dissolved in dil. HCl and the As is distilled as AsH_3, which is collected in I soln. The As content of the soln. is determined by developing the heteropoly blue compound of As with ammonium molybdate and hydrazine sulphate and measuring its absorption at 840 mμ. The method gives recoveries of ≃ 67 per cent. of added As (5 to 10 μg) when applied to eggs and 99 to 104 per cent. recovery of added As (10 μg) when applied to chicken liver. Application to pig liver gave the same order of recovery as chicken liver. G. P. Cook

1932. **Determination of radioactive gold in biological tissue.** L. C. Weiss, A. W. Steers and H. M. Bollinger (*Anal. Chem.*, 1954, **26** [3], 586–587).—To the sample, add 1 ml of an aqua regia soln. containing 2·5 mg of inactive Au and then sufficient conc. HCl to cover the sample. Heat at 100° C for 3 hr., cool and dilute with an equal volume of water. Add 2·5 g of activated C and continue heating for a further ½ hr. Cool and filter through a "rapid" filter-paper, wash the residue until the washings are colourless and transfer them to a borosilicate dish. Ignite at 600° C until little or no C remains. Leach the residue with small portions of aqua regia (with warming) and take an aliquot of the whole of the sample for mounting and counting. The procedure gives 96 to 107 per cent. recovery over the range of 0·2 to 1000 microcuries of added radiogold. The method is applicable to the analysis of various animal organs, no difficulties being encountered with samples ranging from 0·3 to 532 g and from lung tissue to bone and cartilage. G. P. Cook

1933. **Estimation of fluorine in biological material.** P. Venkateswarlu and D. N. Rao (*Anal. Chem.*, 1954, **26** [4], 766–767).—The sample (0·5 g) is submitted to Willard - Winter distillation with H_2SO_4; to the very slightly alkaline distillate at 100° C is added light MgO (0·2 g), boiling being continued for ≃ 5 min. The MgO (containing adsorbed F) is separated by centrifugation or filtration and is then distilled with $HClO_4$ in presence of barium and silver perchlorates (to retain SO_4'' and Cl'), the fluoride in the distillate being titrated according to the Smith - Gardner procedure (U.S. Atomic Energy Communication, 1948, Unclassified Document UR 36.) The F content of a sample of tea determined as above was 180 p.p.m. in comparison with 30 to 120 p.p.m. obtained by the usual ashing method, which leads to loss of F as iron fluoride. W. J. Baker

1934. **Some improvements in the chromatographic separation of amino-acids with the aid of Dowex 50 ion exchanger.** P. C. van der Schaaf and T. H. J. Huisman (*Chem. Weekbl.*, 1954, **50** [15], 273–275).—The amount of ninhydrin used for each fraction can be reduced from 40 mg to 20 mg, heating being then continued for 30 min. in a water-bath. For the separation of basic amino-acids in urine, it is advisable to use a column of 0·9 × 30 cm. This gives a good separation of histidine and lysine, but there are three components on the histidine spot (including methylhistidine) and more than one on the lysine spot. The pH is important in the ninhydrin reaction, and a change in value from 5·0 to 4·3 decreases the apparent recovery of all amino-acids by about 50 per cent. The pH should be kept between 4·8 and 5·2. By decreasing the grain size of the exchange resin, separation was not improved. G. Middleton

1935. **Quantitative determination of amino-acids using mono-dimensional paper chromatography.** J. F. Roland, jun. and A. M. Gross (*Anal. Chem.*, 1954, **26** [3], 502–505).—Two solvent systems, *sec.*-butanol - 3 per cent. NH_3 (3 + 1) and 72 per cent. phenol, resolve 16 amino-acids well enough for determination by direct photometry of the ninhydrin spots. The alcohol system gives excellent resolution of lysine, arginine, alanine, proline, tyrosine, valine, methionine, *iso*leucine, leucine and phenylalanine. The phenol system will resolve cysteic acid, aspartic acid, glutamic acid, serine, glycine and threonine. For the development of the spots, a soln. of ninhydrin (0·2 per cent.) in acetone containing 1 per cent. of acetic acid is used. The individual estimations of the composition of a 15 amino-acid mixture were in good agreement with the known composition of the mixture, the maximum deviations being given by glutamic acid (— 7·84 per cent.), lysine (+ 9·21 per cent.) and glycine (+ 8·24 per cent.). G. P. Cook

1936. **Chromatographic determination of cystine as cysteic acid.** E. Schram, S. Moore and E. J. Bigwood (*Biochem. J.*, 1954, **57** [1], 33–37).—A method for determination of the cystine plus cysteine content of proteins is described. It is based on oxidation of the protein by performic acid at 0° C, hydrolysis of the oxidised protein by aq. HCl, chromatographic separation of the cysteic acid on Dowex 2 (a basic ion-exchange resin) and elution of the acid with 0·1 *N* chloroacetic acid and its determination in the eluate by the photometric ninhydrin method. The oxidation is effective on insol. proteins, and, under the given conditions, both cystine and cysteine give 90 ± 2 per cent. of the theoretical yield of cysteic acid. The method can be used directly on products in which a high carbohydrate content may make other methods inapplicable. It has been used for analysis of foods and purified proteins. J. N. Ashley

1937. **A modified method for determination of hydroxyproline.** C. J. Martin and A. E. Axelrod (*Proc. Soc. Exp. Biol. Med.*, 1953, **83** [3], 461–462).—The colorimetric method of Neuman and Logan (*Brit. Abstr. C*, 1950, 412) is modified with regard to the removal of excess of peroxide after oxidation of hydroxyproline. Instead of the heating procedure originally used, 0·1 ml of 0·05 *M* $FeSO_4$ containing 0·5 ml of conc. H_2SO_4 per 100 ml is added to each tube, which is shaken for 6 min. This method ensures a linear relation between the colour produced in the final step and concn. of hydroxyproline, and interference by tyrosine is reduced; it is not applicable in the presence of high concn. of tyrosine. H. F. W. KIRKPATRICK

1938. **A simplified photometric analysis for serine and formaldehyde.** W. R. Frisell, L. A. Meech and C. G. MacKenzie (*J. Biol. Chem.*, 1954, **207** [2], 709–716).—A rapid method is described for determination of < 10 μ-mol. of serine wtih an accuracy of $\pm$ 3 per cent. The β-carbon atom of serine is oxidised by HIO_4 to formaldehyde, which is condensed with chromotropic acid to give a coloured product that is determined spectrophotometrically at 570 mμ. In presence of added formaldehyde, serine is determined by difference, without loss of sensitivity or accuracy. The method is applicable to determination of serine and formaldehyde in presence of mitochondrial and liver sediment preparations. Care must be exercised when the method is applied to materials containing dimethylaminoethanol, methylaminoethanol, ethanolamine, glyoxal or threonine, because these compounds give a colour with chromotropic acid after oxidation with HIO_4. Many other biologically important substances do not interfere. J. N. ASHLEY

1939. **A new method for the detection of proteins on paper in paper electrophoresis.** W. F. Bon (*Chem. Weekbl.*, 1954, **50** [8], 131–132).—When a moist strip of paper containing protein bands is pressed against a slightly larger strip of stout silver bromide paper, an image of the bands is transferred to the bromide paper; this image can be made visible by exposure and development. If an ultrafast developer is used, and the image is fixed with conc. potassium metabisulphite soln. when the first traces of blackening appear, the bands are represented by black patches on a white background. When the paper is fully developed with a slow developer, the bands are seen as dark patches on a black background. In an experiment with horse serum on S & S 2043B paper moistened with a barbitone sodium buffer of pH 8·6, a concn. of 1 μg per cu. mm of serum gave a detectable blackening. The bromide papers used were: Gevaert bromide Parix brillant glanzend No. 8, Ilford bromide ultrahard glossy B5.1P and Ilford bromide hard matt B3.55K. E. HAYES

1940. **An improved apparatus for the filter-paper electrophoresis of serum and other proteins.** G. T. Franglen (*J. Clin. Pathol.*, 1953, **6** [3], 183–186).—Constructional details and operational technique are given for a filter-paper electrophoresis apparatus designed to give undistorted protein bands up to a width of 25 cm, to enable a greater range of analyses to be carried out on the separated protein fractions. The test soln. is applied to the paper by means of prepared strips, and the degree of separation attained is independent of the position of application. A constant-flow device enables the range of buffers used to maintain constant pH over a prolonged period to be extended. H. F. W. KIRKPATRICK

1941. **Improved colorimetric determination of urinary 17-ketosteroids.** H. Werbin and Siew Ong (*Anal. Chem.*, 1954, **26** [4], 762–764).—Hydrolysis and extraction of urine and colour development by the Zimmermann reaction are carried out by established methods. For the colour test, the extract and reagents are mixed in a 12-ml test tube, the mouth of which has a standard glass joint that can be fitted into the socket of a 65-ml separator. About 3 min. before the end of the 1 hr. allowed for colour formation, 8·0 ml of anhyd. ether are added from a burette to the separator. At the end of exactly 1 hr. from the addition of the 2·5 *N* alcoholic KOH, 1 ml of water is added to the test tube and the contents are mixed; the test tube is plugged into the separator whilst the latter is held almost horizontal and the contents are shaken vigorously to extract the pink chromogens. The inverted test tube is removed, and, after separation, the aq. layer is discarded. One ml of absolute ethanol is added to the ether soln. and the pink colour is measured at 515 mμ against a reagent blank. A standard curve was prepared with dehydro*epi*androsterone. It is claimed that this method nearly eliminates interfering substances absorbing strongly at 400 mμ and gives results comparable with those given by other methods after Girard separation, hence rendering the Girard separation unnecessary. Recovery of crystalline steroids added to urine extracts averaged 96 per cent. H. F. W. KIRKPATRICK

1942. **New method for rapid chromatographic separation and identification of adrenocortical steroids: application to biological fluids.** M. M. Pechet (*J. Clin. Endocrinol.*, 1953, **13** [12], 1542–1544).—The method requires no pre-impregnation of paper and uses water as the stationary phase. A 3-limb single sheet in descending chromatography (*Science*, 1950, **111**, 6; *J. Biol. Chem.*, 1951, **188**, 763) is used, the paper being Whatman No. 11 and the developing solvent a mixture of toluene (20 vol.), light petroleum (10 vol.), butanol (1·5 vol.), ethanol (1·5 vol.) and water (7 vol). The upper phase of the equilibrium mixture is pipetted into the trough, and the bottom of the chamber is covered, with a mixture of both phases. The paper is removed when the solvent front reaches the bottom of the limb and is air-dried for 10 min. The steroids are detected with alkaline $AgNO_3$ and triphenyltetrazolium chloride. R_F values of $C_{21}O_3$ steroids are greater than those of $C_{21}O_4$ steroids, which in turn are greater than those of $C_{21}O_5$ steroids. Twenty-four hr. urine samples are continuously extracted with ether without pre-acidification; the ether extracts after washing with alkali and H_2O are evaporated *in vacuo* and the residue is partitioned between light petroleum and 70 per cent. ethanol. The aq. ethanol phase is evaporated *in vacuo*, the residue is dissolved in a known vol. of $CHCl_3$ and aliquots are applied to the paper for chromatography. Other aliquots are fractionated with Girard-T compound into ketonic and non-ketonic constituents and chromatographed separately. By this method Compounds E and F were easily detected in the urine of a leukaemic boy treated with ACTH; the amounts were estimated semi-quant. by a knowledge of the limits of sensitivity of the alkaline $AgNO_3$ test applied to serial dilutions. H. F. W. KIRKPATRICK

1943. **Determination of adrenocortical steroids in mixtures.** E. Heftmann and D. F. Johnson (*Anal. Chem.*, 1954, **26** [3], 519–521).—A method for

the separation of six active adrenocortical hormones by partition chromatography on silicic acid columns is given. Partitioning takes place between the stationary water phase and a mobile light petroleum - dichloromethane phase of gradually increasing polarity. The steroids are eluted in the order of increasing polarity and are determined by their absorption at 248 mμ. Identification of the steroids in the various fractions is achieved by the H_2SO_4 method. G. P. Cook

1944. **Sulphuric acid induced fluorescence of corticosteroids.** M. L. Sweat (*Anal. Chem.*, 1954, **26** [4], 773–776).—The following method is used for routine quant. analysis. Set the fluorimeter to zero with a blank tube containing 0·1 ml of redistilled pure ethanol and 1 ml of pure conc. H_2SO_4. To 0·1 ml of pure ethanol containing 0·005 to 30 μg of 17-hydroxycorticosterone, add rapidly 1 ml of conc. H_2SO_4 and mix by thorough stirring with a "footed" glass rod. Read the galvanometer deflection after 20 min. For exciting max. fluorescence, primary filters (Corning 5113 + 4489) limiting the mercury light to the region of 436 mμ were found most effective, and max. galvanometer deflections were given with a combination of Wratten No. 16 and No. 74 as secondary filters. A number of steroids were examined by this technique and the relative fluorescence was recorded. The most active were: 17-hydroxycorticosterone, 100; 4-pregnene-11α:17α:21-triol-3:20-dione, 100; corticosterone, 87; 4-pregnene-11α:21-diol-3:20-dione, 88; 4-pregnene-11β:17α:20β:21-tetrol-3-one, 80; and oestradiol, 106. The method, in conjunction with suitable partitioning techniques to separate the steroids, can be applied to quant. determination of 17-hydroxycorticosterone and corticosterone in biological materials; it is more sensitive than the phenylhydrazine method. For amounts of steroid between 0·05 and 5·0 μg, the relation with galvanometer reading is rectilinear, the optimum range for biological work being 0·25 to 1·25 μg.
H. F. W. Kirkpatrick

1945. **Separation of steroid hormones and their metabolites by partition-type chromatography.** E. R. Katzenellenbogen, K. Dobriner and T. H. Kritchevsky (*J. Biol. Chem.*, 1954, **207** [1], 315–321).—The preparation and use of partition-type chromatographic columns for separation of adrenal and urinary steroids are described. The stationary phase is silica gel impregnated with ethanol or formamide, and the mobile phase is either methylene chloride containing 1 to 5 per cent. of ethanol or *cyclo*hexane or a mixture of *cyclo*hexane and benzene. Steroids with a keto group at C_{11} are easily separated from the 11β-hydroxy analogues. Compounds differing only with respect to the steric configuration of the ring juncture at C_5 or by presence or absence of unsaturation in conjugation with a keto group are also readily separated. 3α:17α-Dihydroxypregnan-17-one and 3α:11β-dihydroxyandrostan-17-one are not resolved on the silica - ethanol column, but they are readily separated from each other, and also from other C_{11} oxygenated 17-ketosteroids on the silica - formamide column. Infra-red spectrometry, supplemented occasionally by paper chromatography and development of a fluorescence with H_2SO_4, is used for identification of the steroids. For determination, u.v. spectrometry is used for compounds that contain the conjugated Δ^4-3-ketone system; the data are calculated with the average value $\epsilon_{240} = 16{,}600$. The 17-ketosteroids are determined by a modified Zimmermann colour reaction. 21-Acetoxy-11β:17α-dihydroxypregnane-3:20-dione, 21-acetoxy-17α-hydroxypregnane-3:11:20-trione, 3α:21-diacetoxy-11β:17α-dihydroxypregnan-20-one and 3α:21-diacetoxy-17α-hydroxypregnane-11:20-dione are determined by measuring in the i.r. the percentage transmission at the max. of the carbonyl stretching absorption characteristic of each compound. The method is reproducible to within ≃ 10 per cent. J. N. Ashley

1946. **A method for the quantitative analysis of 17-ketosteroid mixtures.** B. L. Rubin, R. I. Dorfman and G. Pincus (*J. Biol. Chem.*, 1953, **203** [2], 629–645).—The method described, which involves the Zimmermann reaction, can be used on a semi-micro scale for the determination of urinary components present to the extent of ≃ 100 μg per 24 hr. Paper chromatographic methods in two-solvent systems (heptane - 2-phenoxyethanol and heptane - propylene glycol) are used, and standard conditions are defined for resolution of nine components of the α-ketonic extracts of urine. These are 3β-chloroandrost-5-en-17-one, an unknown steroid, androsterone, aetiocholan-3α-ol-17-one (**I**), 11-ketoandrosterone, aetiocholan-3α-ol-11:17-dione, androst-9(11)-en-3α-ol-17-one (**II**), aetiochol-9(11)-en-3α-ol-17-one (**III**) and an unresolved polar mixture. The least polar compounds are resolved with the heptane - 2-phenoxyethanol system, and the more polar compounds with the other system. These solvent systems do not resolve a mixture of androsterone and **II** or a mixture of **I** with **III**. But the $\Delta^{9(11)}$ unsaturated compounds are oxidised by perbenzoic acid to the respective epoxides, and the mobilities of these in the heptane - propylene glycol system are sufficiently different from the mobilities of androsterone and **I**. The method is applicable to extracts of other body fluids or tissues.
J. N. Ashley

1947. **Reactions of certain unsaturated steroids with acid iron reagent.** B. Zak, N. Moss, A. J. Boyle and A. Zlatkis (*Anal. Chem.*, 1954, **26** [4], 776–777).—Pipette 1·0 ml of steroid soln. (25 mg per cent. w/v in glacial acetic acid) into a test tube, add 2·0 ml of colour reagent (1 ml of 10 per cent. w/v $FeCl_3.6H_2O$ in glacial acetic acid made up to 100 ml with conc. H_2SO_4), allowing it to flow down the side of the tube to form a separate layer. Record the colours of the several rings and then mix by tapping the tube laterally. The colours appear immediately, Δ^4 and $\Delta^{4,7}$ steroids giving a brownish-yellow or purple ring, a thin yellow ring only appearing with saturated compounds. Δ^5, $\Delta^{5,7}$ and $\Delta^{5,16}$ steroids give a multicoloured ring (usually 3 or 4 colours), which may include yellow, red, pink, orange, violet or brown. The colours of the final soln. are stable and can be used for absorptiometric determination of Δ^5 sterols such as cholesterol, pregnenolone, stigmasterol, sitosterol and others.
H. F. W. Kirkpatrick

1948. **Relations between phase composition and partition coefficients for some neutral steroids. A nomographic approach.** L. L. Engel, J. Alexander, P. Carter, J. Elliott and M. Webster (*Anal. Chem.*, 1954, **26** [4], 639–641).—The quarternary two-phase system consisting of ethyl acetate, *cyclo*hexane, ethanol and water was studied for the separation of neutral steroids by counter-current distribution procedures. By appropriate experimental design, the partition coefficients of the solutes over a wide range of phase composition were found

in relatively few determinations. The data were converted to a nomographic form that permits the determination of a partition coefficient over a wide range of phase compositions. Nomograms for androsterone, 5-*iso*androsterone, *epi*androsterone, dehydro*epi*androsterone, androstanedione, Δ^4-androstenedione, deoxycorticosterone and 17-hydroxycorticosterone are given. From these nomograms it is possible to predict the behaviour of the steroids in counter-current distribution experiments and also to select systems suitable for the resolution of mixtures. G. P. COOK

1949. **Measurement of enzyme reaction velocities at low temperatures.** V. P. Maier and A. L. Tappel (*Anal. Chem.*, 1954, **26** [3], 564–566).—The method of determining peroxidase reaction velocities at very low temp. is described to illustrate the application of the direct spectrophotometric method. A spectrophotometer is equipped with thermoplates and apparatus necessary to attain low temp. Various substrate solutions containing guaiacol and H_2O_2 are placed in a cuvette and the enzyme is blown into this soln., the increasing absorbance at 430 mμ being recorded at various time intervals. The effect of temp. on peroxidase reaction velocity in 50 per cent. methanol, 60 per cent. glycerol and in 5 *M* Na acetate is illustrated. G. P. COOK

1950. **Colorimetric micro-determination of cytochrome-c oxidase.** W. Straus (*J. Biol. Chem.*, 1954, **207** [2], 733–743).—A colorimetric method is described for determination of small amounts of cytochrome oxidase. It is based on formation of indophenol blue from 1-naphthol and dimethyl-*p*-phenylenediamine in presence of the oxidase. The dye is extracted with $CHCl_3$ - ethanol (9 + 1), and the intensity is determined on a photo-electric colorimeter. The formation of indophenol blue varies according to the amount of enzyme and also to the time of incubation at 37° C during the first few min. (1 to 5 min.). The method may have to be modified for determination of micro amounts of the enzyme, because with mitochondrial suspensions (as source of enzyme) that contain < 10 μg of N, the formation of the blue colour is not proportional to concn. of enzyme. Proportionality can be restored approximately by decreasing the concn. of the other reagents and by incubation at 0° C for 1 hr. or longer. J. N. ASHLEY

1951. **A spectrophotometric method for the determination of choline dehydrogenase.** H. J. Eichel (*J. Biol. Chem.*, 1954, **207** [2], 647–655).—The method proposed is for assay of choline dehydrogenase activity in rat liver and kidney homogenates and depends on determination of the rate of reduction of added cytochrome-*c*, which is determined spectrophotometrically at 550 mμ. Optimum activity occurs at pH 6·8 and with a final PO_4''' concn. of 0·067 *M*. Addition of diphosphopyridine nucleotide has no effect on the rate of reduction; nicotinamide slightly inhibits reduction of cytochrome-*c*. The amounts of the enzyme in rat liver and kidney are stated, and are compared with those found by the manometric method. J. N. ASHLEY

1952. **The determination of glucosamine.** R. Belcher, A. J. Nutten and C. M. Sambrook (*Analyst*, 1954, **79**, 201–208).—The colorimetric method of Elson *et al.* (*Brit. Abstr. A*, 1934, 175) for determination of glucosamine by reaction with acetylacetone and *p*-dimethylaminobenzaldehyde is critically examined and an improved method is derived. The glucosamine soln. is treated with both reagents according to the procedure described, the optical density of the coloured solution is measured at 512 mμ on a suitable spectrophotometer and the concn. is ascertained from a standard graph. The method has been applied to determination of glucosamine in *N*-acetyl-α-methylglucosamide and heparin. A. O. JONES

1953. **The polarographic determination of 3:3′:5-tri-iodothyronine.** H. E. Evert (*Arch. Biochem. Biophys.*, 1954, **49** [1], 93–97).—3:3′:5-Tri-iodothyronine is determined by the method described for thyroxine by Borrows, Page and Hems (*J. Chem. Soc.*, 1949, 204). A slightly modified base solution is used. W. H. C. SHAW

See also Abstracts 1872, 1955, 1961, 2008, 2018.

Drugs

1954. **Concerning the practicability of the quantitative estimation of santonin in santonica.** H. Geyer (*Dtsch. Apotheker-Z.*, 1954, **94** [17], 338–340).—A review of the relative merits of the published methods for the quant. determination of santonin in drugs. They include gravimetric, volumetric, colorimetric and polarographic methods. 31 references. N. M. WALLER

1955. **On the reliability of the method used in the assay of heparin.** B. Blombäck, M. Blombäck, E. V. Corneliusson and J. E. Jorpes (*J. Pharm. Pharmacol.*, 1953, **5** [12], 1031–1040).—Methods reviewed include: (*i*) the thrombin - plasma method of Studer and Winterstein (*Helv. Physiol. Pharmacol. Acta*, 1950, **9**, 6), (*ii*) the fresh whole-blood method of Quick (*Amer. J. Physiol.*, 1936, **115**, 317), (*iii*) the U.S.P. method by means of citrated plasma plus calcium salt and (*iv*) the B.P. method with sulphated whole-blood plus thrombokinase. Thrombin methods, such as (*i*) in which heparin is neutralised by pre-formed thrombin, are preferred to non-thrombin methods, (*ii*), (*iii*) and (*iv*), which may be influenced by re-calcification, pro-accelerin and proconvertin.

Method (*i*) gave consistent results from day to day; but the authors differed from Studer and Winterstein in the preparation of the plasma, preferring to freeze it immediately after centrifugation. They claimed that the original procedure, in which the plasma was allowed to stand at 0° C for 24 hours before freezing, gave results that were too high. Method (*ii*) is not suitable for accurate analysis and requires some degree of skill in operation. The method of the U.S.P. gave results 10 to 15 per cent. lower than those by the thrombin method. This observation is confirmed by other workers. The B.P. method, although a non-thrombin method, is described as being rapid and accurate. The equipment required is simple and the method has the advantage that the results can be submitted to variance analysis. G. B. CHESHER

1956. **The quantitative determination of penicillin O by infra-red analysis.** W. W. Wright (*Antibiot. & Chemother.*, 1954, **4** [1], 71–75).—The infra-red mull method described by Garlock and Grove (*Brit. Abstr. C*, 1949, 239) can be used for potassium penicillin O (allylthiomethyl penicillin) but not for 2-chloroprocaine penicillin O, because of interference at the wavelengths used. The penicillin O from

the potassium salt (100 mg) or the 2-chloroprocaine salt (168 mg) is extracted from an acid (H_3PO_4) solution of the sample into $CHCl_3$, the infra-red absorption spectrum of the dried $CHCl_3$ extract being recorded on a single-beam spectrometer between 10·7 and 9·4 μ. From observations at 10·1 μ (absorption peak) and 10·3 μ (transmission peak), the optical density is found by means of a base-line technique, and the purity of the sample is calculated by reference to a calibration with standard potassium penicillin O. Results are generally lower than in the infra-red mull method (*loc. cit.*) and have a slightly higher average deviation. Penicillin G does not interfere. W. H. C. SHAW

1957. **The determination of neomycin in mixtures with dihydrostreptomycin.** J. Levine, H. Fischbach and B. Arret (*Antibiot. & Chemother.*, 1954, **4** [3], 266–269).—In the FDA cup-plate assay for neomycin (**I**), the interference of dihydrostreptomycin (**II**) is avoided by preliminary hydrolytic inactivation of **II** with $Ba(OH)_2$.

Procedure for oily preparations—Dissolve 1 ml or 1 g in 50 ml of ether and extract with four 20-ml portions of distilled water. Dilute the combined aq. extracts to 100 ml. Transfer 10 ml (containing $\gtrsim$ 200 μg of **II** per ml) to a 25-ml calibrated flask and add 1 ml of 5 per cent. $Ba(OH)_2.8H_2O$. Heat for 3 hr. in a steam-bath, cool, add 1 drop of 1 per cent. phenolphthalein soln. and neutralise by dropwise addition of *N* H_2SO_4. Dilute to volume with water, centrifuge and decant the clear supernatant liquid. For bio-assay, add, to an appropriate aliquot, sufficient *M* potassium phosphate buffer (pH 8·0) to provide, after dilution to the proper vol. with water, a final solution 0·1 *M* with respect to buffer and containing 10 μg of **I** per ml. Complete the assay by the FDA cup-plate method.

W. H. C. SHAW

1958. **The biologic[al] assay of neomycin in combination with dihydrostreptomycin, using a test organism made resistant to dihydrostreptomycin.** J. C. DeNunzio, F. W. Bowman and A. Kirshbaum (*Antibiot. & Chemother.*, 1954, **4** [3], 300–303).—Abnormally high values are obtained for neomycin (**I**) in the presence of dihydrostreptomycin (**II**) when **I** is determined by the FDA plate assay method by use of *Micrococcus pyogenes* var. *aureus* as test organism. A culture (ATCC 6538P) of the organism was made resistant to concentrations > 2000 μg per ml of **II** by preliminary growth in broths containing increasing amounts of **II** and then by the gradient plate method of Szybalski (*Science*, 1952, **116**, 46). The treated culture maintained sensitivity to **I** and resistance to **II** for at least a month in refrigerated storage. Results for **I** (10 μg per ml) with this culture are accurate in the presence of up to 1000 μg of **II** per ml and compare favourably with those given by bio-assay after inactivation of **II** with 5 per cent. $Ba(OH)_2$ (Levine *et al.*, Abstract 1957 above).

W. H. C. SHAW

1959. **New colorimetric method of estimating propylthiouracil and similar compounds.** F. Reinhardt (*Hoppe-Seyl. Z.*, 1953, **293** [6], 268–272).—A new colour reaction for propylthiouracil and similar compounds is based on the reaction of the thioureido group with $RuCl_3$ in strongly acid solution. The deep red-violet colour, which shows an absorption max. at 520 mμ, is shown to be affected by degree of acidity, temp., time, illumination and concn. of the reagent. The optimum conditions are stated. The reaction can be applied to the detection on paper chromatograms of quantities as small as 1 μg per ml. Derivatives of thiourea and thiobarbituric acid give a similar colour, and mercaptoiminazole gives a blue-green colour.

P. G. MARSHALL

1960. **Qualitative and quantitative analysis of mixtures of sulphonamides. VIII.** P. L. de Reeder (*Anal. Chim. Acta*, 1954, **10** [5], 413–421).—To find the correct end-points for the acidimetric titration of the sulphonamides, the pH values of aq. solutions of their Na salts are determined. The errors arising from the use of unsuitable indicators are discussed and thymolphthalein is recommended as giving results of practical value for mixtures. The $\cdot SO_2NH\cdot$ group can be titrated in *n*-butylamine with 0·1 *N* Na methoxide (Fritz and Lisicki, *Brit. Abstr. C*, 1951, 454) and *p*-nitrophenylazoresorcinol as indicator. Some sulphonamides can be determined by pptg. their Ag salts from neutral or slightly alkaline soln., filtering and titrating the excess of Ag^+ in the filtrate. Some experiments are also described on the hydrolysis of the sulphonamides and the determination of the basic or acidic products of hydrolysis. W. C. JOHNSON

1961. **Analytical problems in the determination of Evans blue [azovan blue] caused by adsorption on glass and protein surfaces.** W. O. Caster, A. B. Simon and W. D. Armstrong (*Anal. Chem.*, 1954, **26** [4], 713–715).—The major problem in adapting Evans blue (azovan blue) methods to the microlitre scale was caused by the tendency of this dye to be adsorbed on glass and protein surfaces. In protein-free soln., as much as 20 to 50 per cent. of the dye is adsorbed on the walls of the cuvette; this figure could be higher under certain conditions of pH and salt. concn. The more acidic the solution the greater the adsorption on glass. This adsorption is reduced at pH 8, but this pH has little effect on the extent of adsorption on plasma protein. At pH > 8·0 there is a shift of absorption max. towards shorter wavelengths as the pH increases. These errors are eliminated by addition of a high concn. of Zephiran (benzalkonium chloride). This detergent prevents adsorption of the dye on glass surfaces and removes it from protein. G. P. COOK

1962. **Titrations in non-aqueous solutions [of acids and bases in tobacco smoke].** B. R. Warner and W. W. Haskell (*Anal. Chem.*, 1954, **26** [4], 770–771).—The methods described are applicable to the determination of total strong and weak acids and bases in tobacco smoke. The first method requires a calomel and a glass electrode, an acetic acid (glacial) bridge being used between the former electrode and the solution to be titrated. This eliminates errors in titration, normally caused by changing liquid junction potential as the KCl flows into the solution being titrated. The titration is carried out in glacial acetic acid or glacial acetic acid - *cyclo*hexane (10 + 5 by vol.) with 0·005 *N* $HClO_4$ in glacial acetic acid as titrant. For the determination of total acidity, an Sb - glass electrode cell with butylamine as solvent is used. The titrant is Na methoxide in 10 per cent. v/v methanolic benzene. The methods differentiate between acids and bases having dissociation constants of the order of 10^{-5} and those having dissociation constants of the order 10^{-9}. G. P. COOK

1963. **The colorimetric determination of "Carbowaxes" and their behaviour with alkaloid reagents.** D. Coppini and R. Cameroni (*Boll. Chim. Farm.*, 1953, **92** [10], 363–367).—The behaviour of Carbowax 1500 and Carbowax 4000 towards 30 reagents

used for testing alkaloids is reported. The Carbowaxes can be determined by treating them with excess of $K_4Fe(CN)_6$ and determining the excess of $K_4Fe(CN)_6$ colorimetrically. For Carbowax 4000, add 1 ml of HCl soln. (20 per cent. v/v) to 1 to 10 ml of a 1 per cent. soln. of the wax; add 3 ml of 0·5 per cent. $K_4Fe(CN)_6$, make up to 20 ml and filter after 20 min. To 2 ml of the filtrate, add 0·5 ml of $FeCl_3$ soln. (0·5 per cent. w/v.) and, after 30 seconds, 0·5 ml of 2 per cent. soln. of crystalline oxalic acid. Make up to 20 ml and, after 30 min., measure the extinction in Pulfrich photometer with a S61 filter against a distilled-water blank. For Carbowax 1500 the method is similar, except that 2 ml of HCl, sp. gr. 1·18, are used, and before the $FeCl_3$ is added, the acidity is reduced by the addition of 2 ml of *N* NaOH soln.; more acid conditions are necessary for the pptn. of the $K_4Fe(CN)_6$ - Carbowax 1500 complex. The method gave good results with suppositories containing barbitone and phenobarbitone in Carbowax bases. E. HAYES

1964. **Systematic toxicological analysis by spectrophotometric methods. I. Non-alkaloidal organic compounds.** G. W. Roche and H. N. Wright (*Arch. Ind. Hyg. Occ. Med.*, 1953, **8** [6], 507–517).—A spectrophotometric method for the qual. identification of the non-volatile non-alkaloidal organic poisons usually encountered in routine toxicological analysis is described. The characteristic u.v. absorption curves (over the range 220 to 320 mμ) of the compounds are presented together with the wavelength of their points of max. absorption and the concn. ranges over which they can be quant. determined. Acetanilide, phenacetin, phenazone, amidopyrine, cinchophen, *neo*cinchophen, the barbiturates, 1-naphthylthiourea, DDT, *o*-nitrophenol, *p*-phenylenediamine, santonin and cinnamic acid can be determined quant., but acetylsalicylic acid, Na salicylate, hexylresorcinol, hydroquinone, 2:4-dinitrophenol, 2:4:6-trinitrophenol, picrotoxin and benzoic acid can only be determined qualitatively. The tissue (5 to 10 g) and McIlvain's buffer (5 to 10 ml) at pH 7 are homogenised for 10 min.; 10 g of the homogenate are extracted with exactly 100 ml of $CHCl_3$, and the solution is evaporated to dryness at 60° C. The residue is taken up in ethanol for the determination. J. M. JACOBS

1965. **Systematic toxicological analysis by spectrophotometric methods. II. Alkaloidal poisons.** E. Berman and H. N. Wright (*Arch. Ind. Hyg. Occ. Med.*, 1953, **8** [6], 518–527).—The method is similar to that described in abstract 1964 above. The sample of tissue is homogenised with 25 ml of 0·1 *N* HCl; the homogenate is extracted on a water-bath with ≃ 75 ml of 95 per cent. ethanol and 2 ml of 10 per cent. Na_2WO_4, the residue being dissolved in 50 ml of McIlvain's buffer at pH 7 and extracted with $CHCl_3$ (50 ml). The separated $CHCl_3$ layer is then extracted with 100 ml of 0·1 *N* HCl. The characteristic u.v. absorption curves for 30 alkaloids in dil. HCl are presented. Strychnine, brucine, atropine, hyoscine, codeine, *apo*morphine, aminophylline, theobromine Ca salicylate, physostigmine, quinine, quinidine and cinchonine can be determined quantitatively by this method. J. M. JACOBS

Food

1966. **Rapid determination of sodium chloride in foodstuffs containing protein. II.** K. W. Gerritsma and J. Willems (*Chem. Weekbl.*, 1954, **50** [15], 271–272).—A modification of the method previously described by the authors (*Brit. Abstr. C*, 1950, 341). The material, such as fish, ham, bread or cheese, is disintegrated with 300 ml of water and 3 ml of pentanol in a high speed mixer (14,000 r.p.m.). After reducing the speed to half, there are added 5 ml of 10·6 per cent. $K_4Fe(CN)_6.6H_2O$, followed after a short time by 5 ml of 23·8 per cent. $(CH_3{\cdot}COO)_2Zn.3H_2O$. After 2 minutes at full speed, the mass is rinsed, made up to 500 ml and filtered; 50 ml of the filtrate are taken for the determination of chloride by the Volhard method. G. MIDDLETON

1967. **Chromatographic analysis of sugars in bread.** T. Griffith and J. A. Johnson (*Cereal Chem.*, 1954, **31** [2], 130–134).—The sugars are extracted from 25 g of bread crumbs by refluxing for 2·5 hr. with 80 ml of ethanol (80 per cent.), the slurry is centrifuged, ethanol is removed from the supernatant liquid under reduced pressure, and the conc. solution is diluted to 25 ml with water and de-ionised batch-wise with 0·5 g each of Dowex 50 and Duolite A_4. The sugars in 5 to 12 μl are separated by chromatography on Whatman No. 1 filter-paper for 39 hr. at 82° F by a mixture of propanol, ethanol and water (7:1:2) as solvent. The sugars are identified by spraying with either aniline - diphenylamine or 3:5-dinitrosalicylic acid soln. and determined by the method of Dubois *et al.* (*Nature*, 1951, **168**, 167). S. C. JOLLY

1968. **Determination of reducing sugars in bread by biological methods.** R. T. Bohn (*Cereal Chem.*, 1954, **31** [2], 87–99).—A method is described for determining hexoses (D-glucose and D-fructose) and maltose in bread crumbs, without preliminary extraction, by measuring manometrically the respective vol. of CO_2 produced on fermentation with bakers' yeast and with a special yeast incapable of fermenting maltose. Lactose is determined gravimetrically by the usual methods after removal of the other sugars by fermentation. S. C. JOLLY

1969. **A rapid method for the determination of cereal lipase activity.** R. B. Koch, A. R. Felsher, T. H. Burton and R. A. Larsen (*Cereal Chem.*, 1954, **31** [2] 113–120).—The method is based on electrometric titration of free fatty acids liberated from a buffered emulsified substrate. To 4·0 g of defatted wheat germ dispersed in 100 ml of water at 36° C, 10 ml of buffer solution (phosphate or borate) at pH 7·6 are added, followed by 10 ml of butter-fat emulsion stabilised with 3 per cent. of a soya-bean phospholipid (Aztec 4135). During the ensuing 60 min. at 36° C, the pH of the continuously stirred reaction mixture is maintained at 7·6 ± 0·02, by the addition of 0·1 *N* NaOH, and the titre is plotted against time, zero time for the reaction being taken as 10 min. after addition of the emulsion. A blank determination is made by heating the wheat germ and water for 1 hr. in a bath of boiling water and cooling to 36° C before adding the buffer and emulsion. S. C. JOLLY

1970. **Modified Amdur's dipicrylaminate method applied to the study of potassium contents of maple syrups.** E. Bois and M. Jean (*Anal. Chem.*, 1954, **26** [4], 727–730).—Add 5 g of syrup (of known solid content) to a platinum dish and heat overnight at 100° C. Treat the hardened syrup with a drop of olive oil and heat for 30 min. under an i.r. lamp and then ignite at 550° C until the ashes are white. Cool, add 1 or 2 ml of water, dry under the i.r. heater and complete the calcination in the same

furnace. Take up the ashes with water at 100° C, cool and make up to 25 ml. Pour this soln. into a test tube, add 0·1 g of MgO and heat to incipient boiling. To a 1-ml aliquot of this soln., add 1 ml of Li dipicrylaminate, mix thoroughly and set aside overnight at room temp. Pour the liquid into a standardised Kahn tube, centrifuge and measure the absorption at 595 mμ. Prepare a calibration curve, using solutions equivalent to 0 to 1·0 mg of K_2O in 1 ml of water, proceeding from the point where 1 ml of the Li reagent is added. The reproducibility is 0·01 per cent. of the K found, expressed on a dry basis. G. P. COOK

1971. **Chromatographic determination of mannose.** H. Sobue, K. Matsuzaki and A. Hatano (*J. Soc. Text. Cellulose Ind. Japan*, 1953, **9** [11], 565–567).—Four methods of determining mannose (boric acid method, paper chromatography, chromatopile and phenylhydrazone method) are compared. The boric acid and the chromatographic methods are the most accurate, the minor components causing no interference. Mannan was detected in the turbid fraction of high-grade cellulose acetate pulp, but its presence does not cause the same degree of turbidity as pentosan. R. B. CLARKE

1972. **Ultra-violet spectrophotometry. I. Estimation of theobromine and caffeine in cocoa products. II. Spectrophotometric titrations [involving the dichromate ion].** J. W. Miles (*Dissert. Abstr.*, 1954, **14** [2], 247).—**I.** Theobromine is isolated from cocoa powder (0·3 g) by aq. extraction (Dekker batch-method) followed by clarification with $Zn_2Fe(CN)_6$ and adsorption on columns of Fuller's earth. After elution from the column by NaOH, the theobromine is estimated by u.v. spectrophotometry of the acidified soln. Caffeine from a 1-g sample is estimated similarly in a $CHCl_3$ extract of the $Zn_2Fe(CN)_6$ filtrate made alkaline with aq. NH_3. **II.** Solutions containing Fe^{II} (< 30 mg) are titrated with $K_2Cr_2O_7$ at 350 mμ in presence of H_3PO_4 to complex the Fe^{III}; those containing < 0·2 mg of Cr (as $K_2Cr_2O_7$) are titrated with $FeSO_4$ at 310 mμ; the absorption of the Fe^{III} complex is used to follow the titration. Solutions, such as those met with in steel analysis, containing < 20 mg of Cr (as $K_2Cr_2O_7$) and < 0·5 mg of V (as vanadate) are titrated first with H_3AsO_3 and then with $FeSO_4$. A quartz titration cell in conjunction with a Beckman spectrophotometer is used; the mean error is ± 0·3 to ± 0·6 per cent. W. J. BAKER

1973. **Determination of acidity of wines.** J. Štefanić (*Mitt. Wein u. Obstbau, Wien*, 1954, **4** [3], 134–139).—For routine determinations of pH values of wine (accurate to ± 0·03), two quinhydrone electrodes are suspended, one in a buffer soln. of Na_2HPO_4 plus citric acid at pH 4 and the other in the sample; the electrodes are connected through a galvanometer, and the buffer soln. is titrated with 0·1 *N* HCl until the electrodes are at the same potential, a capillary electrometer being substituted for the galvanometer towards the end of the titration. A table is given for the conversion of amount of 0·1 *N* HCl used to pH (range 4·00 to 2·60). Total acid contents (accurate to ± 1·5 per cent.) are similarly determined, except that a buffer soln. of pH 7·0 is used, the wine is titrated with standard alkali soln. and the galvanometer is used throughout the titration. The buffering capacities of four wines were determined by noting changes in pH produced by additions of known amounts of $CaCO_3$ or tartaric acid; the results obtained were not adequately explained by relations between the contents of acid and those of ethanol extract, or ash (or its alkalinity). The buffering capacity of wine might prove a useful datum. P. S. ARUP

1974. **Contribution to the study of vanilla extracts; new methods for the determination of vanillin.** A. Maurel (*Compt. Rend. Acad. Agric. France*, 1954, **40** [4], 163–165).—The requisite properties of commercial vanilla extracts are discussed, and two methods for the determination of vanillin are described. One utilises the reaction of the phenolic grouping with nitrous acid to give the yellow nitroso deriv. Nitrosation is effected on a 10 per cent. vanilla soln. at pH 4, clarified with Pb acetate, excess of Pb being removed with H_2SO_4. The deriv. of vanillin and diethylvanillin give a specific max. absorption at 415 mμ. The second method is based on oxime formation, a potentiometric titration determining the HCl liberated when vanillin and hydroxylamine hydrochloride react together. The methods are convenient and give results in accordance with the standard methods. N. M. WALLER

1975. **Comparison of the characteristics of vegetable oils extracted with various chlorinated solvents.** G. Jacini, C. Carola and K. Giese (*Olii Grass. Sap.*, 1954, **31** [3], 39–40).—Yields, acidities, contents of impurities, unsaponifiable matter and hydroxy-acids and transmittances at various wavelengths are recorded for arachis, rape, sunflower, linseed, tomato-seed, castor, soya bean, grape seed, linseed cake and olive pulp oils after extraction with hexane, trichloroethane, dichloroethane and dichloromethane, the last of these in general having the highest selectivity next to that of hexane. A special study is made of the extraction of olive oil, which has the lowest acidity when extracted with trichloroethane. R. C. MURRAY

1976. **A quick dilatometric method for control and study of plastic fats.** N. D. Fulton, E. S. Lutton and R. L. Wille (*J. Amer. Oil Chem. Soc.*, 1954, **31** [3], 98–103).—A dilatometric method is described for the study of fat phase behaviour particularly for shortening and margarine. The instrument is of volumetric type with Hg as confining fluid. From a given equation, the "Solid Content Index," which is an approximation to the true percentage of solid, is found. The method is rapid (owing to the introduction of a "tempering" period to expedite approach to a steady state before final measurement), consistent and reliable, and can be applied to basic study of composition and processing and to hydrogenation control. D. BAILEY

1977. **Estimating carbonyl compounds in rancid fats and foods.** A. S. Henick, M. F. Benca and J. H. Mitchell, jun. (*J. Amer. Oil Chem. Soc.*, 1954, **31** [3], 88–91).—A modified method for the quant. determination of the carbonyl compounds in fats and oils is described. The procedure is based on the formation of the 2:4-dinitrophenylhydrazones of the carbonyl compounds in the presence of trichloroacetic acid as catalyst and on the colorimetric determination of the hydrazone compounds in alkaline soln. A benzene soln. of the fat is added to benzene solutions of trichloroacetic acid (4·3 per cent.) and 2:4-dinitrophenylhydrazine (0·05 per cent.) and after heating (60° C, 30 min.), the mixture is treated with 4 per cent. alcoholic KOH and diluted to a standard volume with ethanol. After

10 min. the absorbances are determined at 430 and 460 mμ against a blank. Standardisations are presented for the simultaneous determination of saturated and allenic carbonyl content from the absorbances at 430 and 460 mμ. Applications of these procedures to edible oils are described.

D. BAILEY

1978. **The chemical determination of vitamins A and D with electrophilic metal halides. III. The determination of vitamin A with $FeCl_3$.** J. Brüggemann, W. Krauss and J. Tiews (*Z. anal. Chem.*, 1954, **141** [3], 161–173).—An extensive theoretical treatment of the reaction of Carr - Price reagent with vitamin A leads a technique that gives a stable colour. By using $FeCl_3$, which is insoluble in chloroform, the vitamin A is first converted to polyenes with five or six conjugated double bonds; acetic anhydride is added and the $FeCl_3$, which then dissolves in the chloroform layer, reacts with the polyene to give a stable blue colour with maxima at 570 and 620 mμ. To the chloroform soln. of vitamin A (2 ml), add the reagent solution (0·03 ml), made by adding 2 ml of acetyl chloride to 2 ml of a 5 per cent. soln. of $FeCl_3.6H_2O$ in conc. HCl, and swirl for 20 seconds; add acetic anhydride (0·03 ml), shake the soln. for 30 seconds and measure the extinctions at 570 and 620 mμ and add these two values. Construct a calibration graph in a similar manner and calculate the concn. of the unknown.

P. S. STROSS

1979. **The chemical determination of vitamins A and D with electrophilic metal halides. IV. Recommendations for the chemical determination of vitamin A in biological materials by use of electrophilic metal halides.** J. Brüggeman, W. Krauss and J. Tiews (*Z. anal. Chem.*, 1954, **141** [3], 173–182).—The various stages in the preparation of biological material, feeding stuffs, oils, etc., for the assay by the modified Carr - Price reagent previously described (*Anal. Abstr.*, 1954, **1**, 1978) are critically examined. Chromatography is generally unnecessary even for substances such as margarine containing little vitamin A. A typical example is the assay of vitamin A in rat liver. To finely cut liver (1 to 3 g) in an open evaporating basin, add 1 per cent. alcoholic hydroquinone soln. (1 ml) and 5 per cent. alcoholic KOH soln. (10 to 15 ml), and evaporate to a thick syrup at 80° C. Add activated alumina (8 ml), mix well and transfer to a column. Elute the vitamin A with ethanol and water-free chloroform (20 to 30 ml) and determine the vitamin A by the modified Carr - Price reaction with $FeCl_3$.

P. S. STROSS

1980. **The detection of the vitamins of the B group by means of small circular chromatograms.** A. Jones, M. P. Taylor and D. N. Gore (*Chem. & Ind.*, 1954, [16], 461–462).—Nicotinic acid or nicotinamide (0·01 μg) was detected by chromatographing their solutions on circular filter-papers, placing these on the surface of nutrient agar seeded with *L. arabinosus*, and incubating at 30° to 37° C for 4½ hr. or longer.

M. TADMAN

1981. **Biological determination of vitamin B_{12} with *Euglena gracilis*.** J. Janicki, J. Pawelkiewicz, S. Stawicki and K. Zochrow (*Przem. Chem.*, 1953, **32** [12], 614–616).—A strain of *Euglena gracilis* was grown on a nutrient free from vitamin B_{12}; this was used to determine the vitamin B_{12} in substances derived from the cultivation of *Adinimycetes* and various bacteria. The results agree within ± 5 per cent. with those obtained by the spectrophotometric method. The biological method is troublesome, but there is no substitute for it in the analysis of fermented substances or materials of a complicated chemical nature.

H. BURSTIN

See also Abstracts 1869, 1933.

Sanitation

1982. **Determination of low concentrations of carbon dioxide in water.** J. B. Smith, E. K. Gilbert and M. P. Howie (*Anal. Chem.*, 1954, **26** [4], 667–670).—In the modified gas-evolution method described, CO_2 is removed from the acidified heated sample (≃ 3 litres) by a stream of recirculating air (provided by a diaphragm-pump in closed circuit) and is then absorbed in 0·03 *N* $Ba(OH)_2$, the excess of which is titrated with 0·04 *N* HCl. Data on a number of variables involved in the analysis are reported and discussed. The constant error is calculated as ≃ 0·045 p.p.m. of CO_2 for concn. ⩾ 1·5 p.p.m. The procedure is applicable to feed-waters from which CO_2 has been removed by a de-aerating heater or demineralising plant to give final concn. of < 0·04 p.p.m.

W. J. BAKER

1983. **Determination of chlorine in water. Suggested use of 3:3′-dimethylnaphthidine.** R. Belcher, A. J. Nutten and W. I. Stephen (*Anal. Chem.*, 1954, **26** [4], 772–773).—Of a number of amines tested, 3:3′-dimethylnaphthidine is most sensitive as regards formation and stability of a coloured product in presence of oxidising agents; it is recommended for the colorimetric detection and determination of 0·05 to 1 p.p.m. of Cl in H_2O. To the sample (100 ml) is added 1 ml of a 0·1 per cent. soln. of the amine in glacial acetic acid; ≃ 5 min. after mixing the soln. thoroughly, the intensity of colour is measured photo-electrically by means of a green filter and compared with standard calibration curves. Acetate buffers and mineral acids should be absent; oxidants other than Cl are generally removed by the procedures used in water analysis.

W. J. BAKER

1984. **Determining residual chlorine in water by neutral o-tolidine methods.** A. T. Palin (*Wat. & Sewage Wks.*, 1954, **101** [2], 74–76).—In this progress report, full details are given of the Palin method for the determination of free chlorine, chloramine, dichloramine and nitrogen trichloride in water. Minor modifications are mentioned.

A. WEBSTER

1985. **The determination of the nitrite content of water with the photo-electric colorimeter.** R. Sijderius (*Chem. Weekbl.*, 1954, **50** [4], 56–58).—A study is made of the method of Shinn (*Ind. Eng. Chem., Anal. Ed.*, 1941, **13**, 33) in which the diazo compound formed by the reaction of nitrite with sulphanilamide is coupled with N-(1-naphthyl)-ethylenediamine dihydrochloride to give a red colour. For NO_2' concn. of 0·002 to 1·000 mg per litre Beer's law is obeyed; there is no difference between extinction measurements made 15 min. and 3 hr. after the colour formation, but after 19 hr. values are lower. The proportions of the reagents may be varied within fairly wide limits without affecting the results. The sulphanilamide reacts completely in 1 min., and extension of the reaction time to 10 min. makes no change. The reaction between ammonium sulphamate and nitrite is also complete in 1 min. In the coupling reaction 10 min.

is required to develop the colour fully. Except for $Fe^{\cdots}$, metal ions do not interfere; addition of 10 mg of $Fe^{\cdots}$ to a soln. containing 0·1 mg of NO_2' in 1 litre increased the extinction at 550 mμ from 0·408 to 0·463. The method of Shinn is considered to be superior to the Griess - Romijn method in which 1-naphthylamine and sulphanilic acid are used to develop the colour. E. HAYES

1986. **Micro-method for determining organic carbon in water containing chlorides.** H. Kay (*Chem.-Ing.-Tech.*, 1954, **26** [3], 156).—Organic C in sea water is determined by wet combustion with H_2SO_4, containing potassium and silver dichromates, in an electrically heated (130° C) and completely closed vessel into which the sample (5 to 10 ml containing 20 to 400 μg of C) and the acid are measured through a two-way tap. A current of O carries the evolved gases through aq. KI, over metallic Sb, through a combustion tube where the oxidation of CO is completed on a platinum contact and SO_2 is absorbed by Ag, and finally into a titration vessel connected to burettes containing 0·05 *N* $Ba(OH)_2$ and 0·025 *N* HCl. There is no access of air during the entire operation. Dissolved CO_2 is removed from the acidified sample *in vacuo* before the test. A. R. PEARSON

1987. **Spectrophotometric determination of uric acid [in water] and some redeterminations of its solubility.** D. R. Norton, M. A. Plunkett and F. A. Richards (*Anal. Chem.*, 1954, **26** [3], 454–457).—The method is based upon the reaction of uric acid with arsenotungstophosphoric acid reagent in the presence of CN′. The absorption of the blue colour formed is measured at 890 mμ. The colour development is controlled by the pH, the best results being obtained at pH 8·1; the time for max. absorption varies between 10 and 60 min. depending on conditions. The average deviation of the method is < 2 per cent. and in a 20-ml aliquot of a natural water sample, uric acid concn. of 0·1 μg per ml can be measured. The method is specific for natural waters. The solubility of uric acid in distilled water is 5·1 mg per 100 ml at 30·2° C; in sea water having a chlorinity of 20·21 g per kg and a pH of 8·29 it is 35 mg per 100 ml at 30·2° C. G. P. COOK

1988. **Further experiences with the bromothymol-blue - glucose - agar plate in routine examination of water.** A. Broscheit (*Z. Hyg. InfectKr.*, 1953, **136**, 575–578).—As a result of previous investigations, tests for acid-forming bacteria by the bromothymol-blue - glucose - agar plate method have been used along with bacterial counts and tests for *Bacterium coli* in the routine examination of water samples. Results are given and discussed of tests for acid-formers in 1 ml and *Bact. coli* in 10 ml, on samples from small towns, villages, individual supplies and proposed supplies. Of 862 samples, 420 were negative and 253 positive in both tests; 130 samples were negative for *Bact. coli* and positive for acid-formers; 59 samples were positive for *Bact. coli* and negative for acid-formers. The causes of the discrepancies are discussed. The conclusions drawn are that the presence or absence of acid-formers is a more valuable indication of the suitability of a water for supply than the total bacterial count, and that the test for acid-formers along with the test for *Bact. coli* is suitable for confirming and supplementing conclusions on the quality of water supplies.
WATER POLLUTION ABSTR.

1989. **The magnetic oxygen analyser in studies of oxygen uptake.** S. R. Hoover, L. Jasewicz and M. Porges (*Wat. & Sewage Wks.*, 1954, **101** [2], 81–83).—The magnetic oxygen analyser devised by Pauling *et al.* (*Brit. Abstr. C*, 1946, **314**) has proved useful in following the course of aerobic oxidation systems and the action of the air dispensing systems of sewage plants. The range of the instrument (100 to 160 mm) covers O removals of 0·33 per cent. at ordinary atm. pressures with an accuracy of ± 0·1 per cent of O. A. WEBSTER

1990. **Quantitative determination and metabolism of hexachlorocyclohexane.** K. van Asperen and F. J. Oppenoorsh (*Chem. Weekbl.*, 1954, **50** [20], 353–356).—The colorimetric determination of hexachlorocyclohexane (**I**) by the Schechter and Hornstein method (*Anal. Chem.*, 1952, **24**, 544), carried out by means of the apparatus described by Reith (*Anal. Abstr.*, 1954, **1**, 160), was found suitable for quant. determination of the γ-isomer of **I** in animal material. After administration to mice, very low concn. of the γ-isomer were found in the brain, liver and kidneys even when the concn. in other organs were high. Both in mice and **I**-resistant house flies, a rapid breakdown of the administered **I** takes place; the amounts excreted are negligibly small.
A. STORFER

1991. **Infra-red spectroscopic method for the quantitative analysis of multi-component systems, as exemplified by the isomers of hexachlorocyclohexane.** R. Mecke and R. Mutter (*Z. Elektrochem.* 1954, **58** [1], 1–8).—An equation is devised for calculating the proportions of α- and γ-$C_6H_6Cl_6$ in a mixture from the intensities of the i.r. absorption peaks for the overlapping bands of the two isomers and of their point of intersection. A procedure is worked out for analysing the spectra of the technical product containing α-, γ- and δ-$C_6H_6Cl_6$ and other impurities, giving results accurate to ± 2 per cent. for the components, even in the presence of large amounts of impurities. R. C. MURRAY

1992. **Abridged procedure in the Schechter method for analysing DDT residues.** P. L. Pontoriero and J. M. Ginsburg (*J. Econ. Ent.*, 1953, **46**, 903–904).—The sample of DDT-treated material is extracted with a definite vol. of benzene, the extract being decolorised by shaking for 10 min. with a mixture (2 to 3 g per 100 ml) of Filter Cel and carbon (4 + 1) and filtering. An aliquot of the clear liquid is evaporated in a small flask on a steam-bath in a gentle air current. Five ml of a nitrating mixture (equal vol. of H_2SO_4 and HNO_3, sp. gr. 1·5) are then added to the flask, which is immersed in a water-bath heated so that the temp. reaches 85° C in 20 to 30 min. The flask is then placed directly on a steam-bath for a further 30 min. The cooled contents of the flask are washed in a separating funnel, first with 25 ml of cold water then twice with 10 ml of water. Twenty-five ml of benzene are then added and the mixture is shaken vigorously for 1 to 2 min. The aq. layer is removed and the benzene soln. is washed with 10-ml portions of 5 per cent. aq. NaOH until the soln. is colourless, and then further washed twice with 15 ml of saturated aq. NaCl. The benzene layer containing the DDT is filtered through dry absorbent cotton. A 2 to 5-ml aliquot is mixed with 5 ml of a Na methoxide solution (10 g in 100 ml of anhyd. methanol) and the blue colour is measured electrophotometricaly after 5 min (λ = 580 mμ). A. G. POLLARD

See also Abstracts 1837, 1998.

Agriculture and Plant Biochemistry

1993. **A rapid method for the determination of nitrogen, phosphorus and potassium in plant materials.** A. J. Cavell (*J. Sci. Food Agric.*, 1954, **5** [4], 195–200).—The proposed method has been used to analyse 15 kinds of plant material; the results agree well with those of standard procedures. Recovery of added N, P and K is excellent, and the coefficients of variation for 12 analyses of a sample of lucerne are 1·22 (N), 1·05 (P) and 1·38 (K). Two g of dried plant material are digested with 30 ml of conc. H_2SO_4, 10 g of Na_2SO_4 and 0·1 g of $CuSO_4$, and the soln. is diluted to 250 ml. A 10-ml aliquot is used to determine N as NH_3 in a micro-Kjeldahl distillation apparatus. P is measured colorimetrically in an aliquot of the soln, which is adjusted to be 1·5 *N* with respect to H_2SO_4 and centrifuged. The soln. is mixed with an $(NH_4)_6Mo_7O_{24}$ - NH_4VO_3 reagent and after 5 min. the light transmissions at 400, 440 and 480 mμ are determined. The percentage of P as P_2O_5 is read from a calibration curve. To determine K, 25 ml of the soln are made alkaline with aq. NH_3, the soln. is atomised into a flame photometer and the percentage of K as K_2O is read from a calibration curve. N. M. WALLER

1994. **A rapid method for the determination of copper in plant material.** T. R. Williams and R. R. T. Morgan (*Chem. & Ind.*, 1954, [16], 461).—The proposed method depends upon the formation of a blue copper complex with bis*cyclo*hexanoneoxalyldihydrazone, the colour absorption being measured spectrophotometrically. There is no appreciable interference from other normal constituents of plant material. Recoveries range from 97 to 102 per cent. M. TADMAN

1995. **Determination of molybdenum in plant tissue.** C. M. Johnson and T. H. Arkley (*Anal. Chem.*, 1954, **26** [3], 572–574).—To a soln. containing 0·1 to 100 μg of Mo is added 10 ml of 6·5 *N* HCl containing 0·05 per cent. of $FeCl_3.6H_2O$, the mixture is diluted to 45 ml and extracted with 3·0 ml of a (1 + 1) mixture of CCl_4 and *iso*pentanol. One ml of 40 per cent. aq. KCNS and 1·0 ml of 40 per cent. $SnCl_2.2H_2O$ in 1·3 *N* HCl are added to the aq. phase and the mixture is extracted with 1·00 ml of the CCl_4 - *iso*pentanol mixture. With precautions to ensure no transfer of water, the organic layer is transferred to a spectrophotometer cell and the absorption is measured at 470 mμ with reference to water; comparison is made with standards that have been treated similarly. Figures for precision are given for a range of Mo concn.
D. A. PANTONY

1996. **Analytical studies on the carbohydrates of grasses and clovers. IV. Further developments in the methods of estimation of mono-, di- and oligosaccharides and fructosan.** C. B. Wylam (*J. Sci. Food Agric.*, 1954, **5** [4], 167–172).—A method is described for the determination of mono-, di- and oligosaccharides and fructosan in fresh and ensiled grasses. The material is extracted with 80 per cent. ethanol for 7 hr. in a Soxhlet extractor and an aliquot of the extract equiv. to 1 g of dry material is used. To this 25 mg of xylose or ribose are added and the soln. is evaporated, clarified with $CdSO_4$ and $Ba(OH)_2$ soln., and de-ionised by electrodialysis or an ion-exchange resin. The glucose, fructose and xylose are separated by paper chromatography, eluted and determined by the Somogyi method. The oligosaccharides remaining on the chromatogram are extracted and are determined after hydrolysis with 0·5 *N* H_2SO_4. N. M. WALLER

1997. **Detection of steroidal pseudosapogenins by infra-red spectroscopy.** A. L. Hayden, P. B. Smeltzer and I. Scheer (*Anal. Chem.*, 1954, **26** [3], 550–552).—Pseudosapogenins possess a band giving 20 to 50 per cent. absorption (under the experimental conditions described) near 1695 cm^{-1} and do not exhibit strong absorption in the 1000 to 800-cm^{-1} region. This band is absent in the original sapogenins, dihydrosapogenins and products resulting from oxidation or reduction at the C_{20}:C_{22} double bond. Various pseudosapogenins were studied in $CHCl_3$ and CS_2 media and as liquid paraffin suspensions or liquid smears; 12 of these i.r. spectra are illustrated. Identification of an individual pseudosapogenin is made by comparison with reference spectra. G. P. COOK

1998. **Estimation of micro quantities of pyrethroids.** A. A. Schreiber and D. B. McClellan (*Anal. Chem.*, 1954, **26** [3], 604–607).—Three methods are given for the determination of allethrin or pyrethrin in solutions, in flour and in impregnated paper or cloth bags. Before the determination, the pyrethroids (chrysanthemumic and pyrethric esters of alkyl*cyclo*pentenolones) are extracted from the flour by light petroleum extraction and from the paper by *n*-butanol extraction. The solutions are then refluxed with 0·1 *N* ethanolic NaOH (3 to 5 ml), $BaCl_2$ soln. (10 per cent.) is added and the soln. is filtered after it has settled. The filter is washed with water and the combined filtrates are acidified with H_2SO_4. Filtration is once more carried out through a Celite-coated paper and the filtrate is extracted with light petroleum; the extracts are evaporated almost to dryness, and the remaining soln. is transferred to a colorimeter tube. The light petroleum is evaporated and 5 ml of yellow HgO reagent (1 g in 120 ml of 33 per cent. H_2SO_4) are added and the reading is taken in a Klett - Summerson colorimeter with No. 54 filters. A calibration curve is prepared with chrysanthemummonocarboxylic acid. G. P. COOK

1999. **The determination of soil pH with the glass electrode and an improved type of Morton cell.** N. Trinder, F. W. Handley and E. M. Drummond (*Analyst*, 1954, **79**, 242–243).—In the modified Morton cell (Gallenkamp's "Laboratory Equipment and Scientific Apparatus," Twelfth Edition, Vol. I, 1951, p. 226), the bridge connection between the two half-cells is formed by a capillary of saturated KCl solution across a three-position ground-in stopcock. Frequent use of the stop-cock for flushing and cleaning of the glass-electrode half-cell caused channelling of the capillary by the soil particles and leakage of the KCl into the glass-electrode cell, thereby causing inaccuracy. In the improved type of cell proposed, the capillary is replaced by a sintered-glass disc of porosity 4 and an agar plug, and the ground-in tap by a flushing arrangement governed by a spring clip. The cell appears to last indefinitely, the limiting factor being the life of the glass electrode. Check tests against the older type of cell showed excellent agreement. A. O. JONES

2000. **Polarographic method for determining total cation-exchange capacity of soils.** K. R. Holtzinger, J. R. McHenry and D. W. Rhodes (*Soil Sci.*, 1954, **77**, 137–142).—The sample (4 g) of air-dried soil is shaken with *N* $MnCl_2$ (33 ml) for 5 min. in a 50-ml

centrifuge tube. After leaving overnight, the mixture is centrifuged (2000 to 2200 r.p.m.) and the supernatant liquid is discarded. The process is repeated three times, centrifugation being applied immediately after shaking. The residual soil is washed three times by centrifugation with 33-ml portions of 95 per cent. ethanol. The absorbed Mn is removed from the soil by centrifuging with three 33-ml portions of *N* KCl, the supernatant liquid being collected and diluted to 250 ml with *N* KCl. A 20-ml aliquot is used for the polarographic determination of Mn after addition of 5 drops of 2 per cent. aq. gelatin as a maximum suppressor, dissolved O being removed by a current of N.

A. G. POLLARD

See also Abstracts 1842, 1881, 1889.

5.—GENERAL TECHNIQUE AND LABORATORY APPARATUS

General

2001. **Testing a quick-weighing balance.** T. W. Lashof and L. B. Macurdy (*Anal. Chem.*, 1954, **26** [4], 707–712).—Tests for determining the linearity of the direct-reading scale of a quick-weighing balance and for calibrating dial operated weights are described. A. J. MEE

2002. **Apparatus for stirring a reaction mixture through a vapour-tight seal.** J. R. Skidmore (*Anal. Chem.*, 1954, **26** [4], 789–790).—A spherical glass joint that rotates not only about the axis of rotation of the stirrer but also about the centre of the spherical joint is used. The stirring rod thus describes a cone, and its zone of influence is large.

A. J. MEE

2003. **Areometry and tensiometry; the present position.** A. Pochan (*Chim. Anal.*, 1953, **35** [12], 332–340).—Densitometers have unequal scale divisions, but measure density directly; areometers have equal scale divisions calibrated to scales associated with Gay-Lussac, Baumé, and so on. The theory of these instruments is discussed in detail followed by a consideration of refinements necessary for accurate work. New pieces of apparatus are reviewed including semi-automatic "areoscopes" and micro-areometers. New apparatus for determining surface tension is also described critically. W. C. WAKE

2004. **High-vacuum fractionation with falling-stream still.** D. J. Trevoy and W. A. Torpey (*Anal. Chem.*, 1954, **26** [3], 492–494).—A falling-stream still - tensimeter fed by a simple pot molecular still is used for the analysis of mixtures of high boiling oils of comparable mol.wt. As little as 0·3 per cent. contamination of di-2-ethylhexyl sebacate with di-2-ethylhexyl suberate can be detected. It is suggested that measurements of evaporation rate in the described apparatus and refractive index are complementary in assessing purity of such oils.

D. A. PANTONY

2005. **High-efficiency still for pure water.** R. Ballentine (*Anal. Chem.*, 1954, **26** [3], 549–550).—A semi-automatic high-efficiency still with a thermal efficiency of 92 per cent. and output of $> 1{\cdot}5$ litres per hr. is described. Contamination of the condensate with metal ions is avoided.

D. A. PANTONY

2006. **Small-scale units for organic identifications.** J. T. Stock and M. A. Fill (*J. Chem. Educ.*, 1954, **31** [3], 144–148).—Details and drawings are given showing the set-up of a compact block-type unit for experienced workers and a more flexible unit for students; they contain the whole of the 28 pieces of apparatus and ancillaries needed for organic identifications. Typical procedures involving identification of hydrocarbons, halides and ethers as their sulphonamides are described. G. HELMS

2007. **Three-level capillary viscometer for study of non-Newtonian flow in sealed systems.** J. C. Honig and C. R. Singleterry (*Anal. Chem.*, 1954, **26** [4], 677–680).—A viscometer designed for the study of the rheological properties of dispersions of certain oil-soluble soaps in non-polar solvents is described. The instrument must exclude moisture and atmospheric O for lengthy intervals, give reproducible results over a 250-fold viscosity range and detect variations from pure Newtonian flow. The absolute dimensions of the instrument are obtained from X-ray photographs and by direct measurement. The instrument can be used to determine shearing stress and rate in absolute units. The treatment of rheological data for non-Newtonian systems is discussed. Rheological properties can usually be specified by two constants, one describing the consistency level of the system under specified shearing conditions and the other characterising deviation from Newtonian flow. The method of obtaining these constants from an equation fitting the flow data is illustrated for typical results.

A. J. MEE

2008. **Oxidation - reduction potential and pH changes in manometric vessels. Method for continuous recording.** K. Wight and D. Burk (*Anal. Chem.*, 1954, **26** [3], 481–484).—A modified Warburg vessel and the attendant electrical measuring circuit, which allows for simultaneous continuous recording of redox potential, pH and pressure changes in a chemical or biological system, are described. The apparatus is used for experiments on ascites tumor, *Chlamydomonas mōwusii* and *Escherichia coli* on various substrates. D. A. PANTONY

2009. **Manometric method for study of solid - gas reactions at moderate temperatures.** P. Souter, J. R. Willimot and T. G. Hunter (*Anal. Chem.*, 1954, **26** [3], 484–487).—A modified Warburg apparatus for determination of pressure changes is used for the measurement of the rate of uptake of O by powdered coal (200 to 230 mesh, 0·2 to 0·8 g) at 94·3° C; probable error is $\pm$ 3 per cent. D. A. PANTONY

2010. **Apparatus for the determination of minor components of a gas mixture.** M. Shepherd (*J. Res. Nat. Bur. Stand.*, 1954, **52** [1], 1–6).—The apparatus is designed to estimate $< 0{\cdot}5$ per cent. concn. of components in a gas mixture without appreciable sorption and desorption of major components in the reagents used to remove the minor ones. Small amounts of solid and liquid absorbents are placed in 4 or 5 suitably designed reaction tubes (2-mm bore, vol. 3·5 ml) connected to and integrated with the distributor of a gas-analysis system, the sample being simply passed from the burette through the tube and into a reservoir over Hg. A description is given of the arrangements and procedures for using the apparatus as a separate unit for effecting complete absorption - combustion analyses or for adapting it to a standard volumetric gas-analysis system. Analyses should be made with dry gases or a

saturator (rather than a desiccant) should be inserted in the reaction tube nearest to the burette.
W. J. BAKER

2011. **Acoustic apparatus for determination of mixture ratio by analysis of engine exhaust gas.** E. F. Weller (*Anal. Chem.*, 1954, **26** [3], 488–491).—Exhaust gas from internal combustion engines is oxidised in a furnace containing Cu - CuO and passed into an analyser that measures the velocity of sound in the CO_2 - N mixture. The described circuit allows conversion of this figure to a d.c. voltage, which is a direct measure of the gas composition. Response time is 30 sec.; probable error is ± 1 per cent.
D. A. PANTONY

2012. **A technique for analysis by counter-current extraction.** M. A. Cookson and D. H. Laney (*Chem. & Ind.*, 1954, [16], 450–452).—A simple apparatus for counter-current distribution of a limited number of transfers comprising essentially six separating funnels attached to a frame that is movable about a horizontal axis, is described and illustrated. A distribution curve for commercial glycerol mono-oleate is shown.
M. TADMAN

2013. **Automatic drive for counter-current distribution apparatus.** E. S. Perry and W. H. Weber (*Anal. Chem.*, 1954, **26** [3], 498–502).—Complete details are given for the fabrication of an automatic drive for counter-current distribution apparatus.
G. P. COOK

2014. **Use of toggle valves and Teflon in hydrocarbon analysis.** R. A. Brown and D. J. Skahan (*Anal. Chem.*, 1954, **26** [4], 788–789).—In the determination of the C_1 to C_5 components of liquid hydrocarbons by the mass spectrometer, it is essential to have representative samples. To eliminate cross contamination of samples, which usually arises through sorption of hydrocarbons in stopcock grease, a manifold is used having no glass stopcocks. A small needle toggle valve is used with a Teflon seating. Glass-to-metal connection was made by Teflon packing glands.
A. J. MEE

2015. **Apparatus for the determination of the pressures of saturated vapours by a static method.** M. Bukala, M. Majewski and W. Rodzinski (*Przem. Chem.*, 1953, **32** [11], 564–568).—The vapour pressure is measured with the aid of a liquid manometer filled with any suitable liquid substance; it forms a separate unit and is connected by a system of glass tubes and cocks with the evaporation section of the apparatus. Any number of measurements can be carried out at various temperatures with one filling of the substance examined (5 ml). The apparatus can be used for the determination of vapour pressures of up to 1200 mm of mercury. Vapour pressure curves are presented for $CHCl_3$ and CCl_4, and the data obtained are collated with those given in the International Critical Tables. At most temperatures the agreement is within 2 to 3 per cent.
H. BURSTIN

2016. **Apparatus for measuring the moisture content of fabrics [such as tyre cord fabrics].** Firestone Tyre and Rubber Co., Ltd., and Jack Bertram Collins (Brit. Pat. 706,487, Date Appl. 22.1.51).—The tyre cord fabric (after dipping in an aq. dispersion of rubber and drying) is passed over an earthed roll and around a charged roll connected to one pole of a ripple-free d.c. source. The arrangement is such that one pole of the d.c. source is connected through a length of the fabric to earth while the other pole of the d.c. source is connected to earth through a known resistance. The p.d. across this resistance gives a measure of the leakage from the source through the fabric to earth and hence of the moisture content of the fabric.
J. M. JACOBS

Optical

2017. **Stray light in a spectrophotometer caused by internal reflection in a 60° prism.** H. D. Einhorn and A. E. Z. Cohen (*J. Opt. Soc. Amer.*, 1954, **44** [3], 232–233).—One stray-light effect in a single monochromator system is due to internal reflection from the walls of a 60° prism. This appears as a "ghost" line at the spectrum position of minimum deviation and amounts in intensity to about 0·1 per cent. of the total energy in the spectrum. In spectrophotometric work, errors caused by this internal reflection can be eliminated by avoiding measurement at exact minimum deviation setting or by using a polarising filter.
B. S. COOPER

2018. **A sensitive photo-electric colorimeter.** H. F. Holden (*Aust. J. Exp. Biol.*, 1953, **31** [2], 173–174).—A photo-electric colorimeter is described having an optical path through the liquid of 7·7 cm, but requiring only 2 ml of soln.; it can be used for small volumes of very pale liquids or determinations in which economy of material has precedence over rapidity. It is especially suitable for determining the dyes adsorbed by proteins in paper electrophoresis.
N. E.

2019. **The design and construction of a differential refractometer for measurements on coloured solutions.** E. Atherton and E. Cowgill (*J. Soc. Dyers Col.*, 1954, **70** [3], 116–120).—The instrument, designed particularly for measurement of refractive indices of dye solutions, is of the prism type, a small spectrometer providing monochromatic light that is concentrated to a narrow beam and passed through the solution near the refracting angle of the prism. The measurements are made by means of a rotating mirror driven by a differential screw, which cancels the deviation of the beam caused by the dye solution. The degree of reproducibility is approx. 2 per cent. in Δn (increase in refractive index due to solute). Graphs of Δn plotted against wavelength are given.
M. TADMAN

2020. **A semi-automatic recording densitometer for use after paper-strip electrophoresis.** A. L. Latner, L. Molyneux and J. Dudfield Rose (*J. Lab. Clin. Med.*, 1954, **43** [1], 157–164).—A detailed description is given of an instrument for the rapid and accurate scanning of paper strips after electrophoresis. The use of the instrument in the analysis of serum proteins is described and, by the technique given, a linear relationship is attained between protein concentration and pen deflection. The instrument is used with reflected light, but it can be converted easily for transmission scanning.
W. H. C. SHAW

2021. **Simple high temperature microscope.** A. Dietzel, O. W. Flörke and H. Saalfeld (*Ber. dtsch. keram. Ges.*, 1954, **31** [3], 80–81).—The specimen is enclosed in a small vertical tube electric furnace and is illuminated by a filament or arc lamp (for the highest temp.), the beam from which is directed by a concave and a plane mirror (Rh on vitreous silica) above the furnace. The reflected light is directed

by the plane mirror through a central hole in the concave mirror and is received by the microscope and 9-cm × 12-cm camera. A magnification of 200 times is achieved. The crystallisation of $8PbO.P_2O_5$ at 860° C is illustrated by photographs at magnifications of 50 to 150. J. A. SUGDEN

Thermal

2022. **Liquid-in-glass thermometers and their use for precision temperature measurement.** A. E. Carte (*S. Afr. Ind. Chem.*, 1954, **8** [3], 50–52).—The characteristics, limitations and methods in use of liquid-in-glass thermometers are described, and details are given of the thermometer standardisation service of the South African National Physical Laboratory. Special reference is made to the use of partial-immersion, Beckmann and spirit thermometers. G. C. JONES

2023. **Improved fractional melting apparatus.** S. V. R. Mastrangelo and J. G. Aston (*Anal. Chem.*, 1954, **26** [4], 764–766).—An improved fractional melting apparatus incorporating compressible conduction vanes is described. It is sturdier than others described previously. Tests of the performance of the apparatus with and without use of the vanes to press out the molten substance are described. The purification efficiency is increased by a factor of three at the 99·98 per cent. level of purity when the compressible vane system is used. A. J. MEE

2024. **An instrument and method for rapid dependable determination of freezing-point depression.** R. L. Bowman, H. V. Trantham and P. A. Caulfield (*J. Lab. Clin. Med.*, 1954, **43** [2], 310–315).—One-ml samples of aq. soln. are frozen in 2 min. by air from a ½ h.p. compressor passing through a Ranque - Hilsch vortex tube system (dimensions are not given). A thermistor-bridge null detector circuit operating at 60 c.p.s. is used for m.p. determinations that are reproducible to within 0·0036° C. Changes of frequency reduce the gain of the amplifier. J. GREEN

Electrical

2025. **Electronic controlled-potential reduction or oxidation apparatus.** F. Kaufman, E. Ossofsky and H. J. Cook (*Anal. Chem.*, 1954, **26** [3], 516–519).—The electrical circuit for an apparatus that allows electrolytic oxidation or reduction to be carried out at const. potential or current is described. D. A. PANTONY

2026. **Measurement of redox potentials.** K. Edelmann (*Faserforsch. u. Textiltech.*, 1953, **4**, 337–342).—Apparatus for measuring single redox potentials and studying changes in potential during chemical reactions is described. R. B. CLARKE

2027. **Electrolyte bridge and reference electrode for potentiometric titrations.** G. Brunisholz (*Anal. Chim. Acta*, 1954, **10** [5], 470–473).—Platinum, silver - silver chloride, and calomel electrodes each of which is fitted into an outer tube by a standard ground-glass joint at its upper end are described. In the tube, the electrode is immersed in an appropriate soln. A liquid junction with the test soln. is established through a hole near the lower end of the tube and this hole is covered with a ground-glass sleeve. W. C. JOHNSON

2028. **Rotated and stationary platinum wire electrodes. Residual current - voltage curves and dissolution patterns in supporting electrolytes.** I. M. Kolthoff and N. Tanaka (*Anal. Chem.*, 1954, **26** [4], 632–636).—Polarograms observed at solid wire electrodes in the presence of non-oxidising or reducing supporting electrolytes often exhibit small cathodic or anodic currents that may interfere with the evaluation of cathodic and anodic diffusion currents. Polarograms obtained with a clean platinum electrode in solutions of pH 1 to 13 in absence of O gave a small anodic pre-wave when measured from negative to positive, the starting point of which varied with pH. Polarograms in the opposite direction from clean electrodes did not show an abnormality, provided the electrode was not polarised at a more positive potential than one corresponding to the beginning of the appearance of the pre-wave. The behaviour of anodically and cathodically polarised electrodes was investigated. Polarograms beginning at the evolution of O to more negative potentials show a small cathodic dissolution pattern starting at a potential that corresponds approximately with the equilibrium value of the platinum - platinum hydroxide electrode. The limiting current of the anodic wave decreases rapidly to zero at a given potential, indicating the formation of a film on the surface of the electrode. A similar film is formed by placing the electrode in solutions of strong oxidising agents. The film can be quickly removed by treatment with $FeSO_4$, or more slowly with As^{III} solutions. The film was not formed when the clean electrode was in contact with O-saturated solutions, but it was formed in O-free solutions of oxidising agents with high oxidation potentials. The formation of the film accounts for the poorly defined anodic diffusion currents often observed at the rotated platinum electrode with automatic recording, and for the fact that the abnormalities on anodic current - voltage curves are not observed when the measurements are made from strongly positive to more negative potentials provided that time is allowed for the formation of the oxide film at the start. A. J. MEE

2029. **The derived polarogram.** H. L. Kies and H. Nijon (*Anal. Chim. Acta*, 1953, **9** [5], 462–466).—The derivative circuit of Heyrovský (*Anal. Chim. Acta*, 1948, **2**, 536; *Analyst*, 1947, **72**, 229) has been modified by the inclusion of a differential galvanometer so that the cathode current flows through one of its two coils. Sensitivity is regulated in each circuit by means of an Ayrton shunt, and the two electrodes are synchronised by the method of Airey and Smales (*Analyst*, 1950, **75**, 287). The relation between max. height and concn. was found to be non-linear, so (in practice) a calibration curve is used. H. F. W. KIRKPATRICK

2030. **Polarographic studies with gold, graphite and platinum electrodes.** S. Lord, jun., and L. B. Rogers (*Anal. Chem.*, 1954, **26** [2], 284–295).—Polarographic waves with gold electrodes were almost indistinguishable from those attained with platinum. Those attained with graphite were sometimes less clearly defined, especially when the electrodes were rotated, but a stationary graphite electrode offered advantages over both gold and platinum ones for studying organic oxidations in which inert non-conducting films are formed. It is known that electrode pre-treatment, rotation, type of electrode reaction and direction of polarisation affect the $E_{\frac{1}{4}}$ value and the diffusion current, so these factors were systematically examined in detail. Results

from ion - ion reactions are compared with those involving formation of a deposit, and results from rapidly reversible reactions are compared with those from slowly reversible or irreversible reactions, The following inorganic reactions have been selected: reduction of $Ag^{\cdot}$ (to exemplify a reversible deposition reaction), oxidation of $Fe^{\cdot\cdot}$ to $Fe^{\cdot\cdot\cdot}$ (for the reversible ion - ion reaction), the reduction of $Fe^{\cdot\cdot\cdot}$ in H_2SO_4 (as a less rapidly reversible reaction), the reduction of $KMnO_4$ to K_2MnO_4 in alkaline soln. (another ion - ion reaction) and the oxidation of NH_2OH (an ion - ion reaction requiring $H^{\cdot}$). In the organic field the moderately rapid oxidation of hydroquinone was chosen to compare with the irreversible oxidation of aniline. In addition brief studies were made on *o*-, *m*- and *p*-phenylenediamine and *o*-, *m*- and *p*-toluidine to obtain potentials to compare with Fieser's critical oxidation potentials (*J. Amer. Chem. Soc.*, 1930, **52**, 5204). Full details of the electrode treatment are given together with polarograms and comparison tables of $E_{\frac{1}{2}}$ values and diffusion currents. B. LAMB

2031. **High-frequency titrations with a resistance-type instrument.** K. Nakano, R. Hara and K. Yashiro (*Anal. Chem.*, 1954, **26** [4], 636–638).—The performance of a resistance-type high-frequency titration apparatus with acid - base titrations and with reactions involving complex formation has been investigated. With acid - base titrations well-defined equivalence points are obtained in agreement with calculated values. Reproducibility and sensitivity are good. The shape of the titration curves is affected by concn. In very dil. solutions, *e.g.*, 0·00097 *N*, no equivalence point is found. The instrument can also be used to determine the composition of dimethylglyoxime - metal chelates. A. J. MEE

2032. **Differential photometric detection in coulometric titration.** E. N. Wise, P. W. Gilles and C. A. Reynolds, jun. (*Anal. Chem.*, 1954, **26** [4], 779–780).—The coulometric titration of acids and bases with photometric determination of the end-point is complicated, during titration, by the formation of bubbles, which the photometric detector is unable to distinguish from a change in density due to a change in colour of the indicator. To overcome this difficulty a differential or ratio-detecting photometer unit has been designed and constructed. A. J. MEE

2033. **A coulometric coulometer.** V. B. Ehlers and J. W. Sease (*Anal. Chem.*, 1954, **26** [3], 513–516).—An apparatus, with attendant electronic circuit, that allows automatic measurement of < 10 coulombs is described. In an electrolyte containing $Cu^{\cdot\cdot}$, ethanol, H_2SO_4, tartrate and hydrazine, Cu is deposited from the anode on the platinum cathode at < 1 mA per sq. mm. The quantity of electricity used in the deposition is determined by quant. stripping of the Cu; 0·01 coulomb is measured within 0·1 per cent. D. A. PANTONY

See also Abstracts 1906, 1999.

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use in the text of an abbreviation or symbol the word is printed in full.

alternating current	a.c.	micro-litre	μl
ampere	amp.	micron	μ
Ångstrom unit	Å	milliampere	mA
anhydrous	anhyd.	milligram	mg
approximate, -ly	approx.	millilitre	ml
aqueous	aq.	millimetre	mm
atmospher-e, -ic	atm.	millivolt	mV
atomic	at.	minimum	min.
boiling-point	b.p.	minute (time)	min.
British thermal unit	B.Th.U.	molar (concentration)	*M*
calculated	(calc.)	molecul -e, -ar	mol.
calorie (large)	kg-cal.	normal (concentration)	*N*
calorie (small)	g-cal.	number	no.
centimetre	cm	observed	(obs.)
coefficient	coeff.	organic	org.
concentrated	conc.	ounce	oz.
concentration	concn.	part	pt.
constant	const.	patent	pat.
corrected	(corr.)	parts per million	p.p.m.
critical	crit.	per cent. wt. in wt.	per cent. w/w
crystalline	cryst.	per cent. wt. in vol.	per cent. w/v
crystallised	cryst.	per cent. vol. in vol.	per cent. v/v
cubic	cu.	potential difference	p.d.
current density	c.d.	pound	lb
cycles per second	c.p.s.	precipitate	ppt.
decompos -ing, -ition	(decomp.)	precipitated	pptd.
density	ρ	precipitating	pptg.
density, relative	*d* or wt. per ml	precipitation	pptn.
derivative	deriv.	preparation	prep.
dilute	dil.	qualitative, -ly	qual.
direct current	d.c.	quantitative, -ly	quant.
distilled	dist.	recrystallised	recryst.
electromotive force	e.m.f.	refractive index	$n\lambda^t$
electron-volt	eV	relative humidity	R.H.
equivalent	equiv.	revolutions per minute	r.p.m.
experiment, -al	expt.	saponification value	sap. val.
gram	g	saturated calomel electrode	S.C.E.
gram-molecule	mole	second (time)	sec.
half-wave potential	$E_{\frac{1}{2}}$	soluble	sol.
horse-power	h.p.	solution	soln.
hour	hr.	specific gravity	sp. gr.
hydrogen ion concentration	[H·]	specific rotation	$[\alpha]\lambda^t$
hydrogen ion exponent	pH	square centimetre	sq. cm
inch	in.	standard temperature and pressure	s.t.p.
indefinite	indef.	temperature	temp.
infra-red	i.r.	ultra-violet	u.v.
insoluble	insol.	vapour density	v.d.
kilogram	kg	vapour pressure	v.p.
kilovolt	kV	volt	V
kilowatt	kW	volume	vol.
liquid	liq.	watt	W
maxim -um, -a	max.	wavelength	λ
melting-point	m.p.	weight	wt.
microgram	μg		

In addition the following symbols are used—

greater than	>	less than	<
not greater than	≯	not less than	≮
is proportional to	∝	of the order of, approximately	≃

The principal Pharmacopoeias are denoted by B.P., U.S.P., or D.A.B., together with the identifying numeral.

Radicles are represented by the usual symbols; positive ions have superscript dots and negative ions superscript dashes, *e.g.*, $Cu^{\cdot\cdot}$, $Al^{\cdot\cdot\cdot}$, Cl', SO_4''. Metals that exist in more than one valency state are represented by their symbols with appropriate superscript roman numerals, *e.g.*, ferric iron becomes Fe^{III} and cuprous copper Cu^{I}.

Volume 1. No. 8, Abstracts 1748-2033 August, 1954

ANALYTICAL ABSTRACTS

A PUBLICATION OF
THE SOCIETY FOR ANALYTICAL CHEMISTRY

CONTENTS

Printed and Published for the Society for Analytical Chemistry by W. Heffer & Sons Ltd., Cambridge, England. Communications to be addressed to the Editor, Norman Evers, 20, Eastcheap, London, E.C.3. Enquiries about advertisements should be addressed to Walter Judd Ltd., 47, Gresham Street, London, E.C.2.

Entered as Second Class at New York, U.S.A., Post Office